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THE BRITISH JOURNAL OF METALS

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INCORPORATING THE METALLURGICAL ENGINEER

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CONTRIBUTIONS

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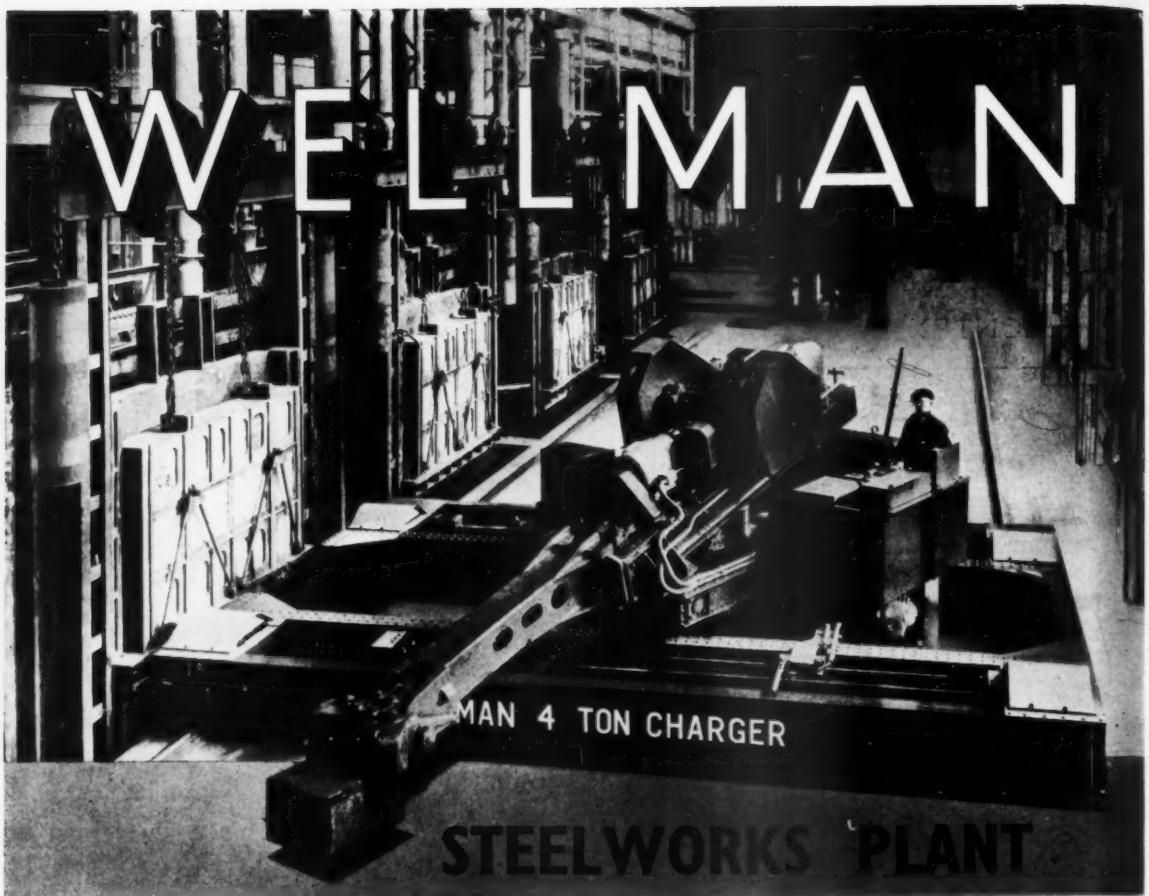

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METALLURGIA

THE BRITISH JOURNAL OF METALS

INCORPORATING THE "METALLURGICAL ENGINEER"

MARCH, 1958 Vol. LVII. No. 341

Safety First

A POSTER which appeared on a number of bill-posting hoardings a few months ago quoted Sir Miles Thomas as saying: "One of the greatest menaces to our industrial health is the noisy worship of ca'canny." Whatever else Sir Miles meant, he was certainly not decrying the adoption of "safety first" practices in industry as a means of cutting down accidents, whether of the lost-time variety or otherwise. No sane industrialist would do so in the face of the simple fact that every day two men are killed and six hundred injured in British factories. In other words, three in every hundred men employed in Britain's industries will have an accident in any one year, and if all these accidents were equally shared out among all workers, no man would go through his working life without at least one accident, and it would be a matter of luck whether that accident were serious or trivial. The economic price of this is twenty million working days lost each year and sixty thousand men absent each day: no one can calculate the cost in human suffering.

Moving from the general to the particular, the iron and steel industry, in spite of recent improvement, still has a total of seventeen thousand lost time accidents a year, which means that—on the average—every person employed in the industry today will meet with an accident at some time or other during the next sixteen years if the accident rate remains at its present level. The key phrase there is "on the average," for there can be little doubt that many individuals will go through their working lives without a lost time accident, whether as a result of being lucky or of being safety conscious. But that only means that someone else is likely to have two, three, or even more, accidents before he retires. So that again one comes to the fact that—in spite of the tendency to talk about the problem in terms of statistics—accidents happen to human beings not to numbers.

This is one of the many aspects of the subject which will be discussed in the new magazine, *Safety*, to be published thrice yearly by the Accident Prevention Committee of the British Iron and Steel Federation. Since its establishment in 1954, the Committee has undertaken a considerable amount of work in the collection of industry-wide statistics and in conducting technical investigations, reports of which have been widely circulated in the industry: it has also encouraged accident research by the British Iron and Steel Research Association. At a conference arranged by the Committee in 1956, a great number of suggestions were put forward by the managers and works safety officers who were present, and some 70% of these suggestions indicated that widespread and regular interchange of information within the industry would prove to be one of the most effective means of reducing the rate of accidents.

It is for this reason that the new magazine has been introduced. Designed to appeal to all levels in the industry, it will contain feature articles, reports and reviews, correspondence, and information on a wide variety of subjects connected with accident prevention and industrial health. Future issues will not be confined to examples from the United Kingdom iron and steel industry, but will include reference to the work of other countries and other industries, as well as safety hazards in the home. It is intended to have a wide circulation in the industry to managers, foremen, safety officers, trade union officials and members of works joint safety committees. Twenty thousand copies of the first issue have been printed, to allow one for every fifteen persons employed in the industry. Copies are also available for interested persons outside the industry.

The new magazine is not of itself going to cut down accidents in a spectacular manner, but inasmuch as it indicates that there are more ways than one of tackling the problem, provides information on the effectiveness of certain measures, and creates that atmosphere of safety consciousness so essential to success, it will be a worthwhile project, and will inspire those who are already keenly interested in safety to fresh efforts to convert the rest of their colleagues to a similar way of thinking. In one iron and steel works, every accident, however trivial, was investigated and action taken to prevent any repetition. As a result, accidents were reduced by two-thirds and man-hours lost fell from fifty thousand to twenty thousand. Such success was not achieved by an isolated safety week, but by a consistent policy designed to make the whole works safety and efficiency conscious. There lies the crux of the problem: as Mr. Richard Summers points out in a foreword to *Safety*, there are many accidents for which there is no obvious cause, where nobody can properly be blamed, but which often are the result of the failure of a man to adjust himself to his environment. The only way of helping him to do this is by proper training and supervision when he enters the industry, for it is well-known that accident-frequency is highest among newly engaged workers, and that as they get more skilled at their jobs they have fewer accidents and less serious ones. At this stage the foremen and charge-hands are the key men, but initial training is insufficient by itself. There must be means by which safety consciousness is maintained, and even increased above this trainee standard. Opposed to the beneficial effect of increased skill, there is the danger of familiarity breeding contempt, which can lead to the most serious accidents of all. It is here that the safety stewards in the various departments can play an important part by pointing out wrong methods and practices on the spot, as they arise, and by setting an example by doing things the safe way all the time. All sections of the industry—management, supervisory staff, safety officers and workers— have a part to play in achieving industrial safety, and truly can it be said that eternal vigilance is the price of safety.

Meeting Diary

11th March

Institution of Engineers and Shipbuilders in Scotland. "Further Applications of Three-Dimensional Extrapolation Methods," by IR. A. J. W. LAP. 39, Elmbank Crescent, Glasgow. 6.30 p.m.

11th and 12th March

Society of Chemical Industry, Corrosion Group. Symposium on the Protection of Motor Vehicles: "Corrosion of Cylinders and Moving Parts of Engines," by W. G. STEVENSON; "Corrosion Inhibitors for Coolant," by A. T. B. P. SQUIRES; "Research and Experience with Sodium Benzoate and Sodium Nitrite Mixtures as Corrosion Inhibitors in Engine Coolants," by A. D. MERCER and F. WORMWELL; "Oil Additives," by N. E. F. HITCHCOCK; "Protection of Accessories," by A. A. TOMPKINS; "Packaging of Vehicles and Components for Export," by R. J. BROWN; "Experience with the Land Rover," by S. HESLOP; "Phosphate Treatment of Vehicle Bodies," by G. MURRAY; "Experience with Aluminium Alloys in Motor Vehicles," by F. C. PORTER. The Institution of Mechanical Engineers, Birdcage Walk, London, S.W.1. 9.30 a.m. and 2.30 p.m.

12th March

Institute of British Foundrymen, Southampton Section. "Fifty Years of Art Founding," by A. L. PARROTT. Technical College, St. Mary's Street, Southampton. 7.30 p.m.

Institute of Fuel, North Western Section. "Applications of the B.P. Corrosion Control Process in Industry," by DR. G. WHITTINGHAM and L. K. KENDALL. Engineers Club, Albert Square, Manchester. 2.30 p.m.

Institute of Metal Finishing, Organic Finshing Group. "Catalytic Combustion of Stoving Oven Exhaust," by A. AIKENS. Royal Society of Tropical Medicine and Hygiene, 26 Portland Place, London, W.1. 6.30 p.m.

Liverpool Metallurgical Society. "Non-Ferrous Tubes in the Stress of Modern Conditions," by C. BRECKON. Liverpool Engineering Society, 9, The Temple, Dale Street, Liverpool. 7 p.m.

Manchester Metallurgical Society. "Martensitic Transformations," by DR. J. W. CHRISTIAN. Manchester Room, Central Library, St. Peter's Square, Manchester, 2. 6.30 p.m.

13th March

Institute of Marine Engineers, North East Coast Section. "Corrosion Control in Tankers," by A. LOGAN, O.B.E. Stephenson Building, King's College, Claremont Road, Newcastle-upon-Tyne. 6.15 p.m. Tea at 5.30 p.m.

Institute of Marine Engineers, West Midlands Section. "Materials for Superheater Tubes and Supports," by D. W. CRANCHER. Birmingham Exchange and Engineering Centre, Stephenson Place, Birmingham. 7 p.m.

14th March

West of Scotland Iron and Steel Institute. "Use and Application of the 'Quantovac'," by S. MUIR. 39, Elmbank Crescent, Glasgow. 6.45 p.m.

17th March

Institute of Metal Finishing, London Branch. "Anodes," by H. CANN. Northampton Polytechnic, St. John Street, London, E.C.1. 6.15 p.m.

18th March

Institute of British Foundrymen, East Anglian Section. "Experiences of Forty Years In and Around Foundries," by W. HARDY. Lecture Hall, Public Library, Ipswich. 7.30 p.m.

Institute of British Foundrymen, Slough Section. Annual General Meeting, followed by a paper on "Non-Destructive Testing of Castings," by S. L. FRY. Lecture Theatre, High Duty Alloys, Ltd., Slough. 7.30 p.m.

Institute of Metals, South Wales Local Section. Annual General Meeting, followed by short papers from members of the Section. Department of Metallurgy, University College, Singleton Park, Swansea. 6.30 p.m.

Institute of Metal Finishing, South-West Branch. "Nickel Plating Techniques," by A. F. BROCKINGTON. Spread Eagle Hotel, Gloucester. 7 p.m.

Sheffield Metallurgical Association. "Trends in Basic Refractories," by H. PARNHAM. B.I.S.R.A., Hoyle Street, Sheffield. 7 p.m.

19th March

Institute of Metal Finishing, Organic Finishing Group. "The Production and Applications of Prepainted Strip," by G. E. BURBAGE. Exchange and Engineering Centre. Stephenson Place, Birmingham. 6.30 p.m.

Powder Metallurgy Joint Group of the Iron and Steel Institute and the Institute of Metals. Informal Discussion (all day) on "Developments in the Practice of Compacting and Sintering." Church House, Great Smith Street, London, S.W.1.

20th March

Incorporated Plant Engineers, London Branch. "The Construction of a Nuclear Power Station." College of Preceptors, Bloomsbury Square, London, W.C.1. 6.30 p.m. for 7 p.m.

Institution of Mining and Metallurgy. "Betterments in the Quality of Refined Lead," by J. S. JACOBI and B. H. WADIA. Geological Society of London, Burlington House, Piccadilly, W.1. 4.30 p.m. for 5 p.m. Visitors will be welcome.

Southampton Metallurgical Society. "Stress Corrosion," by K. WILSON. Southampton University. 7.15 p.m.

21st March

Institution of Mechanical Engineers. James Clayton Lecture: "High-Speed High-Performance Diesel Engines: Their Development and Application," by DR. E. SCHMIDT. 1, Birdcage Walk, Westminster, London, S.W.1. 5.30 p.m. for 6 p.m.

25th March

Sheffield Metallurgical Association. "The Acid Bessemer Process of Today," by F. B. CAWLEY. B.I.S.R.A., Hoyle Street, Sheffield. 7 p.m.

25th–27th March

Institution of Mechanical Engineers. Conference: "Technology of Engineering Manufacture." See programme of Conference for details. Registration required.

26th March

Institute of British Foundrymen, London Branch. "Patternmaking Progress," by B. LEVY. Constitutional Club, Northumberland Avenue, London, W.C.2. 7.30 p.m.

Manchester Metallurgical Society. "Some Metallurgical Aspects of Nuclear Power Development," by DR. H. K. HARDY. Manchester Room, Central Library, Manchester, 2. 6.30 p.m.

28th March

Institution of Mechanical Engineers. "Failure of a 60-MW. Steam Turbine at Uskmouth," by A. L. G. LINDLEY and F. H. S. BROWN. 1, Birdcage Walk, Westminster, London, S.W.1. 5.30 p.m. for 6 p.m.

Institute of Metal Finishing, Sheffield and North-East Branch. "The Analytical Control of Plating Solutions," by K. E. LANGFORD. Fitzwilliam Room, Grand Hotel, Sheffield. 7 p.m.

Society of Chemical Industry, Corrosion Group. Joint Meeting with the Manchester Section. "Recent Research on the Corrosion of Boiler Tubes," by E. C. POTTER. The Robinson Lecture Theatre, The University, Oxford Road, Manchester, 13. 6.30 p.m.

1st April

Incorporated Plant Engineers, Peterborough Branch. "Boiler House Management," by O. H. BARKER. White Lion Hotel, Church Street, Peterborough. 7.30 p.m.

Institution of Engineers and Shipbuilders in Scotland. "Provision of Ore Handling Facilities at General Terminus Quay, Glasgow." 39, Elmbank Crescent, Glasgow. 6.30 p.m.

7th April

Incorporated Plant Engineers, Dundee Branch. "The Uses of Electronics," by R. B. SMITH. Mathers Hotel, Dundee. 7.30 p.m.

10th April

East Midlands Metallurgical Society. Annual General Meeting, followed by "Continuous Rolling of Stainless Steel Strip," by J. A. CAMPBELL. Nottingham and District Technical College, Shakespeare Street, Nottingham. 7.30 p.m.

Institute of Metals, London Local Section. Annual General Meeting, followed by "New Protective Coatings for Metals," by W. E. BALLARD. 17, Belgrave Square, London, S.W.1. 6.30 p.m.

Leeds Metallurgical Society. "Pressure Die Casting," by G. KELLY. West Yorkshire Foundries, Ltd., Leeds. 7.15 p.m.

Liverpool Metallurgical Society. "The Micromechanism of Fracture in Mild Steels," by PROF. W. S. OWEN. Followed by the Annual General Meeting. Liverpool Engineering Society, 9, The Temple, Dale Street, Liverpool. 7 p.m.

The Strain Hardening Behaviour of High Purity Copper

An Appraisal of Tests by Carreker and Hibbard

By E. Voce, Ph.D., M.Sc., F.I.M.
Copper Development Association

SEVERAL years ago Carreker and Hibbard[1] published the results of an extensive series of experiments on the tensile properties of copper of exceptionally high purity. As far as can be judged from subsequent literature, little attention seems to have been paid to this important work, and the aim of the present paper is to bring it to the notice of readers interested in the properties of copper from the practical point of view. Moreover, a critical analysis of the data has enabled certain general conclusions to be drawn concerning the effects of temperature and grain size on the strain hardening properties of copper, and these are thought to be of sufficient value to merit publication.

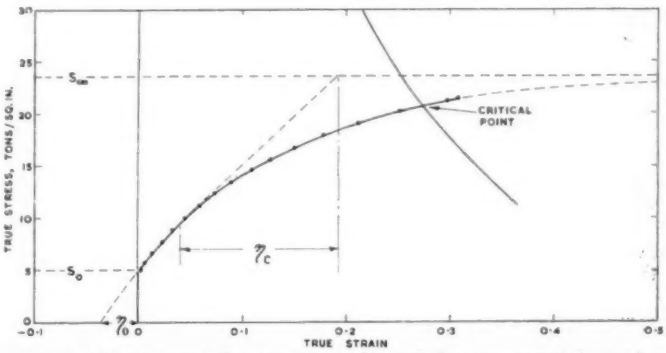

Fig. 1.—Diagrammatic representation of the exponential strain hardening function, based on the test at 0° C. in Table I.

Using annealed copper wire of 99·999% purity, with grain sizes ranging from 0·012 mm. to 0·090 mm., Carreker and Hibbard carried out no less than 40 tensile tests at a series of temperatures between − 253° C. (20° K.) and 950° C. (1,223° K.). The results were presented in the form of true stress-true strain curves, extending in each case up to the point at which necking began and at which the maximum load was attained. The authors also examined the influence of strain rate, but report that its effect was small, especially at low and moderate temperatures. To quote their own words: "... the variation among nominally identical specimens was greater than the effect of strain rate over the ran available."

True stress, S, is defined as the load, P, divided by the sectional area, A, which actually supports it at any instant, and not by the original sectional area, A_o, which is commonly used in ordinary tensile testing procedure.

The conception of true plastic strain was formulated many years ago by Ludwik. An element of true strain, $d\eta$, is the instantaneous change of length, dL, divided by the actual length, L, from which the change takes place, so that

$$d\eta = \frac{dL}{L} \qquad (1)$$

When the strain is zero, the length has its original value L_o, and integration of equation 1 consequently leads to

$$\eta = \log_e \frac{L}{L_o} \qquad (2)$$

During plastic deformation, however, the volume of the specimen remains sensibly constant. That is to say $AL = A_oL_o$, from which it follows that equation 2 can be extended to read

$$\eta = \log_e \frac{L}{L_o} = \log_e \frac{A_o}{A} \qquad (3)$$

Moreover, using the principle of constancy of volume, the true stress can be obtained from an ordinary load-extension diagram, for

$$S = \frac{P}{A} = \frac{P}{A_o} \cdot \frac{A_o}{A} = \frac{P}{A_o} \cdot \frac{L}{L_o} = \frac{P}{A_o} \exp \eta \qquad (4)$$

This method was adopted by Carreker and Hibbard; it is valid up to the point where local necking begins, beyond which the tests were not carried.

Through the courtesy of the authors, open-scale plots of their experimental curves were made available. These were analysed by means of the exponential strain hardening function,[2, 3] namely

$$S = S_\infty - (S_\infty - S_o) \exp\left(-\frac{\eta}{\eta_c}\right) \qquad (5)$$

where S_o, S and η_c are constants, the nature of which will be appreciated from Fig. 1. The two major constants are the *asymptotic stress*, S_∞, towards which the curve trends when the strain becomes indefinitely large, and the *characteristic strain*, η_c, which is the true strain needed to raise the stress from any point on the curve to its asymptotic value along the tangent to the curve at the point in question. The *threshold stress*, S_o, marks the beginning of the curve in any particular circumstances; it has its lowest value when the material is fully annealed, but is raised along the curve by any strain which may have been applied before the test is made. Conversely, the curve may be imagined to extend backwards to stresses below the threshold value, as indicated in Fig. 1.

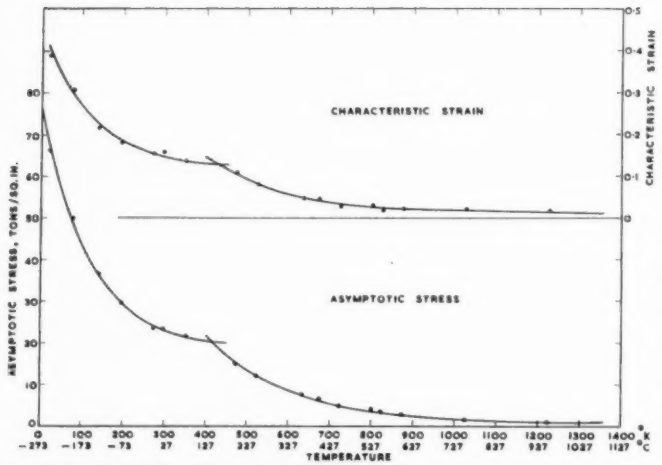

Fig. 2.—Effect of temperature on the average values of the major strain hardening constants.

The *latent strain*, $\eta_\circ$, marked in the diagram, is the strain which would be required to raise the stress from zero to its observed threshold value $S_\circ$. The latent strain can be found by rearranging equation 5, and putting $S = 0$, thus

TABLE I.—EFFECTS OF TEMPERATURE AND GRAIN SIZE ON THE TENSILE STRAIN HARDENING PROPERTIES OF HIGH PURITY COPPER.

Temperature of Test °K.	Temperature of Test °C.	Grain Size (mm.)	Threshold Stress (tons sq. in.)	Asymptotic Stress (tons sq. in.)	Characteristic Strain (logarithmic units)
20	—253	0·012	6·6	71·0	0·445
		0·015	5·0	68·4	0·415
		0·030	3·4	63·9	0·370
		0·045	3·6	62·5	0·364
		0·045	2·9	61·5	0·365
		0·090	3·1	70·0	0·385
78	—195	0·012	6·7	48·6	0·300
		0·015	4·9	46·9	0·287
		0·030	5·1	50·9	0·323
		0·090	3·1	52·6	0·316
140	—133	0·012	5·6	35·5	0·202
		0·045	2·7	37·1	0·237
195	—78	0·012	5·8	29·5	0·175
		0·015	4·9	30·2	0·185
		0·030	4·0	29·5	0·181
273	0	0·012	5·1	23·7	0·152
300	27	0·012	4·9	22·8	0·155
300	27	0·015	4·9	24·6	0·175
298	25	0·030	3·6	23·9	0·159
300	27	0·045	3·1	23·4	0·155
298	25	0·090	2·2	23·2	0·143
350	77	0·015	4·2	21·2	0·142
473	200	0·012	3·8	15·2	0·106
		0·015	4·0	14·7	0·115
		0·030	2·7	15·2	0·113
		0·045	2·0	14·1	0·104
523	250	0·012	3·6	12·1	0·081
631	358	0·015	2·9	7·6	0·048
673	400	0·012	2·25	6·25	0·036
		0·030	2·0	6·25	0·045
		0·045	2·0	6·75	0·053
		0·090	1·8	7·4	0·062
723	450	0·015	2·25	4·9	0·029
800	527	0·012	1·55	4·0	0·032
823	550	0·030	1·65	3·2	0·019
873	600	0·030	1·43	2·63	0·017
		0·045	1·38	2·77	0·022
1023	750	0·045	0·67	1·43	0·022
		0·090	0·77	1·74	0·021
1223	950	0·090	0·36	0·67	0·017

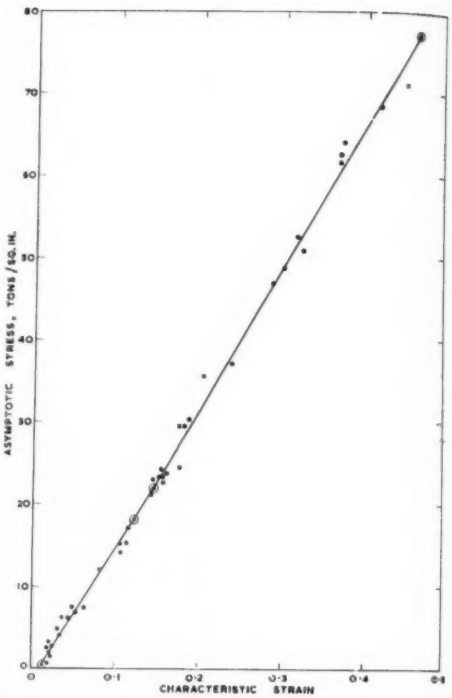

Fig. 3.—Relationship between asymptotic stress and characteristic strain, irrespective of temperature.

$$\eta_\circ = -\eta_c \log_e \frac{S_\infty}{S_\infty - S_\circ} = \eta_c \log_e \left(1 - \frac{S_\circ}{S_\infty}\right) \quad (6)$$

The negative sign merely indicates that the latent strain is measured to the left of the origin, as shown in Fig. 1.

In the discussion which follows, considerable use will be made of the conception of latent strain, especially in connection with the effect of grain size.

The strain hardening constants found for the curves of Carreker and Hibbard are recorded in Table I. The curve shown in Fig. 1 is that for the single experiment at 0° C. (273° K.). The points marked in the diagram are readings taken from the experimental graph, while the curve itself has been drawn from the constants given in Table I for the test in question. The agreement of the equation with the experimental data is clearly satisfactory, and this is equally true for the remaining 39 experiments; Fig. 1 can be regarded as quite typical of them all. The lowermost point lies a little below the curve; this is normal, for it is evidently situated on the small fillet which connects the elastic line with the strain hardening curve.[2, 3]

Examination of Table I shows that all the constants diminish as the temperature is raised. The threshold stress also diminishes consistently as the grain size is increased at any temperature. On the other hand, the major constants show no consistent variation with grain size, although there is considerable scatter at each temperature. It seems legitimate, therefore, to regard the influence of grain size upon the major constants as unimportant, and to consider average values of the asymptotic stress and characteristic strain at each temperature.

These averages are plotted against temperature in

TABLE II.—EFFECT OF TEMPERATURE ON THE ASYMPTOTIC STRESS, CHARACTERISTIC STRAIN AND SPECIFIC WORK OF HIGH PURITY COPPER.

	Asymptotic Stress	Characteristic Strain	Specific Work
Low Temperature Range :			
Initial Value at 0° K.	77 tons/sq. in.	0·46 logarithmic units	17 in. tons/cu. in.
Final Value at High Temperature	18 tons/sq. in.	0·12 logarithmic units	2·9 in. tons/cu. in.
Characteristic Temperature	120° K.	120° K.	120° K.
Activation Energy	238 cal. g. mol.	238 cal. g. mol.	238 cal. g. mol.
High Temperature Range :			
Initial Value at 400° K.	22 tons/sq. in.	0·145 logarithmic units	3·6 in. tons/cu. in.
Final Value at High Temperature	0·5 tons/sq. in.	0·013 logarithmic units	Zero
Characteristic Temperature	200° K.	200° K.	165° K.
Activation Energy	397 cal./g. mol.	397 cal. g. mol.	328 cal./g. mol.

Fig. 2. Each curve consists of two loops, intersecting at a temperature of about 140° C., which is approximately that at which copper of high purity might be expected to anneal during the period of the tensile test. Thus, the low temperature loops represent conditions of cold working, while the high temperature loops are concerned with hot working behaviour.

Each loop of each curve can be represented by an equation identical in form with the strain hardening function, except that the threshold value is greater than the asymptotic value ; thus

$$X = X_\infty + (X_\circ - X_\infty) \exp\left(-\frac{T}{T_c}\right) \qquad (7)$$

where X is the value of either the asymptotic stress or characteristic strain at the absolute temperature T, while the threshold and asymptotic values, $X_\circ$ and X_∞, and the characteristic temperature T_c, are comparable with the corresponding constants in equation 5.

The curves in Fig. 2 have been constructed from the parameters given in Table II, and the fit of the calculated curves to the experimental results is evidently satisfactory. In corresponding loops of the two curves, the characteristic temperature, T_c, is the same. As a consequence, the relationship between asymptotic stress and characteristic strain is linear for each temperature range. From the data in Table II, the straight lines in question are defined by

Low temperature range :

$$S_\infty = 174\ \eta_c - 2{\cdot}8$$

High temperature range :

$$S_\infty = 163\ \eta_c - 1{\cdot}6$$

These lines are shown in Fig. 3, upon which the experimental values of asymptotic stress are plotted against the corresponding values of characteristic strain, irrespective of temperature. The experimental points lie scattered near to the lines.

In dealing with functions like equation 6, it is conventional to convert the characteristic temperature, T_c, to an activation energy, Q, by introducing the gas constant R. On this basis, the equation reads

$$X = X_\infty + (X_\circ - X_\infty) \exp\left(-\frac{RT}{Q}\right)$$

As the gas constant is 1·985 cal./g. mol., the activation energy, expressed in these units is approximately double the characteristic temperature, becoming 238 cal./g. mol. for the low temperature range, and 397 cal./g. mol. for the high temperature range.

It remains to consider the effect of grain size. Table I shows that the smaller the grain size, the higher the threshold stress. Decreasing the grain size is therefore tantamount to applying a small extra latent strain. In a fully annealed pure metal, the most disorganised parts of the lattice are likely to be adjacent to the grain boundaries. In other words, the grain boundaries are probably the most important seat of latent strain. If this is so, there should be a direct connection between the latent strain and the intercrystalline surface area per unit volume. In another context,[4] it has been shown that the intercrystalline surface area per unit volume is inversely proportional to the grain size as normally measured. If, therefore, the latent strain is plotted against the reciprocal of the grain size, the connection just mentioned should be revealed.

To test this view, the latent strain was calculated in accordance with equation 6 for each experiment. The results are set out in Table III. The table is divided horizontally for temperatures within the cold working and hot working ranges respectively, that is to say below and above the intersection of the loops in Fig. 2.

In the lower temperature range, the figures for latent strain depend strongly on grain size, but are substantially independent of temperature. The mean values given in the table are plotted against the reciprocal of the grain size in Fig. 4. The result approximates to a straight line, supporting the inference that the latent strain depends directly on the intercrystalline surface area per unit volume. The equation of the line drawn in Fig. 4 is

$$\eta_\circ = 0{\cdot}013 + \frac{0{\cdot}033}{100\,G}$$

where G is the grain size in millimetres. Zero reciprocal grain size, which gives the initial latent strain of 0·013 logarithmic units, should not be interpreted to mean an indefinitely large single crystal. Rather it represents a normal polycrystalline material in which the intercrystalline surface area is insufficient appreciably to affect the latent strain. A single crystal might well behave quite differently.

TABLE III.—EFFECT OF TEMPERATURE AND GRAIN SIZE ON THE LATENT STRAIN OF HIGH PURITY COPPER.

Temperature		Latent Strain in Logarithmic Units for Stated Grain Size				
° K.	° C.	0·012 mm.	0·015 mm.	0·030 mm.	0·045 mm.	0·090 mm.
20	−253	0·044	0·031	0·021	{0·022 / 0·018}	0·019
78	−195	0·045	0·033	0·034	—	0·019
140	−133	0·035	—	—	0·018	—
195	−78	0·039	0·034	0·027	—	—
273	0	0·037	—	—	—	—
299	26	0·038	0·039	0·026	0·022	0·014
350	77	—	0·032	—	—	—
Mean		0·040	0·034	0·027	0·020	0·017
473	200	0·031	0·037	0·023	0·016	—
523	250	0·029	—	—	—	—
631	358	—	0·023	—	—	—
673	400	0·016	—	0·017	0·019	0·017
723	450	—	0·018	—	—	—
800	527	0·016	—	—	—	—
823	550	—	—	0·014	—	—
873	600	—	—	0·013	0·015	—
1023	750	—	—	—	0·014	0·013
1223	950	—	—	—	—	0·013

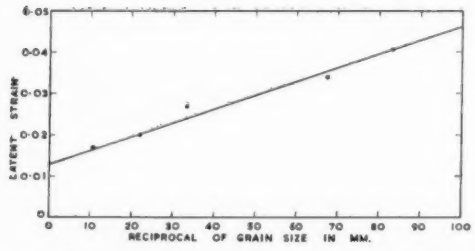

Fig. 4.—Relationship between latent strain and reciprocal grain size, i.e., between latent strain and intercrystalline surface area per unit volume, for the lower temperature range.

Reference to the lower part of Table III shows that all the figures for latent strain tend to diminish as the temperature is raised through the hot working range. The evidence suggests that at high temperatures they would all fall to about 0·013 logarithmic units, which therefore seems to be a basic value for pure copper whenever the influence of grain boundary material is eliminated, either because of large grain size when cold working or by rapid recrystallisation while hot working. At moderate hot working temperatures it is likely that a slow rate of testing would tend to diminish the latent strain towards the basic value by allowing time for recrystallisation.

The foregoing analysis provides a rationalised picture of the effects of temperature and grain size on the mechanical properties of pure copper. It enables the true stress-true strain curve to be constructed with reasonable confidence for any desired temperature and grain size in the cold working range. In the hot working range, the curve can also be derived at any temperature for material having the basic latent strain of about 0·013 logarithmic units. The effect of variations of grain size at moderate hot working temperatures can be roughly assessed by reference to the figures for latent strain in the lower part of Table III, but cannot be predicted with precision because of the incidence of recrystallisation in relation to the testing period. Grain size is not, however, an important factor when hot working, and for most practical purposes it is sufficient to use the basic value for latent strain which applies both to material of large initial grain size and to conditions of full recrystallization.

As an example, a rationalised series of curves has been computed for temperature intervals of 100° C. They are shown in Fig. 5, and the constants on which they are based appear in Table IV. The major constants were derived from the information given in Table II by means of equation 7, and the influence of grain size on the latent strain was assessed from Fig. 4. The corresponding threshold stress was found by using equation 6 inversely. Alternatively, the latent strain can replace the threshold stress in equation 5 by modifying it to read :

$$S = S_\infty \left\{ 1 - \exp\left(- \frac{\eta + \eta_\circ}{\eta_c} \right) \right\} \qquad (8)$$

The curves in Fig. 5 demonstrate the remarkable improvement in the mechanical properties of copper at sub-zero temperatures, a fact brought out in the original paper of Carreker and Hibbard, but which seems to have attracted little attention. Clearly copper is an exceedingly satisfactory material for refrigeration plant, for if designed on the basis of room temperature properties, there will be an increasing margin of safety as the temperature is reduced.

A curve for material of small grain size at 200° C. has been included in the diagram, in spite of the fact that this temperature falls just within the hot working range. High purity copper would recrystallise at 200° C., making the value of the curve questionable, but it would doubtless apply, at least approximately, to copper of more normal purity or to material containing a small addition of silver. Jackson, Hall and Schwope[5] have shown that small variations in the composition of high conductivity copper, including the addition of silver, do not appreciably affect the strain hardening behaviour at room temperature, and the same is probably true at other

TABLE IV.—RATIONALISED STRAIN HARDENING PARAMETERS FOR HIGH PURITY COPPER.

Temperature		Grain Size (mm.)	Primary Constants				Critical Point			Tensile Properties		Specific Work (in. tons/ cu. in.)
°C.	°K.		Latent Strain (logarithmic units)	Threshold Stress (tons/sq. in.)	Asymptotic Stress (tons/sq. in.)	Characteristic Strain (logarithmic units)	Critical Stress (tons/sq. in.)	Critical Strain (logarithmic units)	Critical Work (in. tons/ cu. in.)	Ordinary Tensile Strength (tons/sq. in.)	Limiting Uniform Extension (%)	
−200	73	Large	0·013	2·1	50·1	0·305	38·5	0·434	10·65	25·0	54·2	10·68
		0·04	0·021	3·3	50·1	0·305	38·5	0·426	10·63	25·2	53·1	10·68
		0·02	0·029	4·6	50·1	0·305	38·5	0·418	10·62	25·4	51·9	10·68
		0·01	0·043	6·6	50·1	0·305	38·5	0·404	10·61	25·8	49·7	10·68
−100	173	Large	0·013	2·0	31·9	0·200	26·6	0·346	6·13	22·6	41·2	6·15
		0·04	0·021	3·2	31·9	0·200	26·6	0·338	6·12	22·8	40·1	6·15
		0·02	0·029	4·3	31·9	0·200	26·6	0·330	6·08	23·0	39·0	6·15
		0·01	0·043	6·2	31·9	0·200	26·6	0·316	6·01	23·3	37·2	6·15
0	273	Large	0·013	1·9	24·0	0·155	20·8	0·300	4·27	15·4	35·0	4·29
		0·04	0·021	3·0	24·0	0·155	20·8	0·292	4·25	15·6	33·9	4·29
		0·02	0·029	4·1	24·0	0·155	20·8	0·284	4·22	15·7	32·8	4·29
		0·01	0·043	5·8	24·0	0·155	20·8	0·270	4·17	15·9	31·0	4·29
100	373	Large	0·013	1·8	20·6	0·135	18·2	0·278	3·51	13·8	32·0	3·53
		0·04	0·021	2·9	20·6	0·135	18·2	0·270	3·49	13·9	30·9	3·53
		0·02	0·029	4·0	20·6	0·135	18·2	0·262	3·47	14·0	29·9	3·53
		0·01	0·043	5·6	20·6	0·135	18·2	0·248	3·40	14·2	28·1	3·53
200	473	Large	0·013	1·8	15·5	0·105	14·0	0·232	2·32	11·1	26·1	2·33
		0·01	0·043	5·2	15·5	0·105	14·0	0·202	2·21	11·4	22·3	2·33
300	573	Large	0·013	1·65	9·6	0·069	9·0	0·178	1·20	8·22	19·5	1·21
400	673	,,	0·013	1·45	6·00	0·047	5·74	0·135	0·599	5·01	14·4	0·616
500	773	,,	0·013	1·24	3·82	0·033	3·70	0·101	0·303	3·45	10·6	0·313
600	873	,,	0·013	1·02	2·50	0·025	2·44	0·080	0·165	2·25	8·3	0·171
700	973	,,	0·013	0·80	1·75	0·021	1·69	0·066	0·095	1·59	6·8	0·101
800	1073	,,	0·013	0·66	1·24	0·017	1·22	0·058	0·063	1·15	5·9	0·067

temperatures, apart from the marked influence of purity on recrystallisation. With the latter reservation, therefore, the information provided by Table IV and Fig. 5 can be regarded as an approximate guide to the behaviour of the grades of high conductivity copper in normal commercial production.

Table IV includes information concerning the critical point on each curve at which the maximum load is reached in tension. As explained elsewhere ([2, 3]) this is also the point at which the rate of strain hardening becomes numerically equal to the true stress, and at which the rate of work hardening is unity. Values are given for the critical true stress, the critical true strain and the critical work per unit volume. From them are derived the ordinary tensile strength and the percentage uniform extension before the onset of necking. The final column of the table contains the *specific work*, namely the total input of work required to reduce the rate of work hardening from infinity to unity. This is controlled by the two major constants of the strain hardening function alone, and, like them, is independent of grain size. For the benefit of readers unfamiliar with the subject, the derivation of these various quantities from the primary constants is briefly outlined in an Appendix.

Table IV shows that all the parameters diminish as the temperature is raised. That this is true of the critical strain and its counterpart the limit of uniform extension should not be taken to mean that the overall ductility and malleability of copper is poor at high temperatures, for deformation after the commencement of necking is not covered by the experiments. It does indicate, however, that specimens under tensile, as distinct from compressive, stress will start to fail at high temperatures with comparatively little extension. The limitation is of little practical importance since copper would rarely, if ever, be called upon to carry appreciable tensile loads at high temperatures.

Diminution of grain size at any particular temperature in the cold working range increases the threshold stress and, in a lesser degree, the ordinary tensile strength. The latent strain also increases, but the critical strain falls, so that the sum of the two remains constant. The uniform extension, being closely allied to the critical strain, also falls as the grain size is reduced, and the critical work diminishes very slightly.

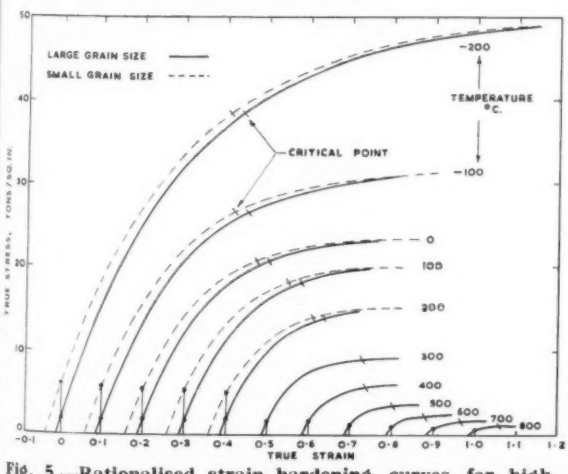

Fig. 5.—Rationalised strain hardening curves for high purity copper, at temperature intervals of 100° C.

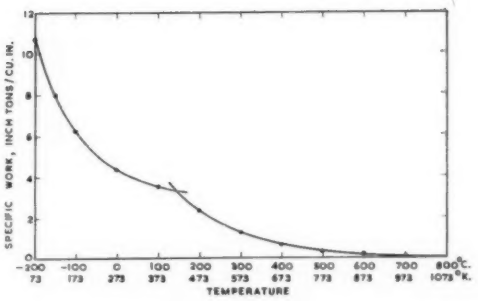

Fig.6.—Effect of temperature on the specific work of high purity copper.

The specific work is necessarily a little greater than the critical work. It reflects the mechanical properties of the material in a particularly fundamental manner, since it expresses the work necessary to change the rate of work hardening from one fixed value, infinity, to another, unity. Thus it enables strict comparisons, embracing both stress and strain, to be made between the results of different tests. The values for specific work given in Table IV are plotted against temperature in Fig. 6. Like Fig. 2, it shows two loops intersecting at a temperature of about 140° C., representing the cold working and hot working ranges respectively. The loops were found to be in good conformity with equation 7, though strictly speaking, they cannot obey this relationship if the asymptotic stress and characteristic strain do so independently. Perhaps an energy relationship such as equation 7 should be more aptly applicable to the specific work than to the individual constants, but this would make interpretation much more difficult, since it would be impossible to separate the influence of stress from that of strain. Whatever the theoretical interpretation, the loops shown in Fig. 6, have been drawn in accordance with equation 7, using the parameters included in Table II, and the fit is clearly satisfactory. For the low temperature range, the characteristic temperature is the same as that for the curves in Fig. 2, namely 120° K., but for the high temperature range it is considerably smaller, being 165° K. instead of 200° K.

In conclusion, it must be emphasised that an analysis such as that described in this paper for high purity copper does not necessarily apply to other materials. There is, for example, some evidence to show that the major constants for 70 : 30 brass are not independent of grain size, as appears to be the case for copper.

APPENDIX

Some Strain Hardening Relationships

By differentiating equation 4 of the text to find $dP/d\eta$, and equating to zero, it can be shown that the maximum load in tension occurs when

$$\frac{dS}{d\eta} = S \qquad (i)$$

That is to say, the critical point is reached when the rate of strain hardening, $dS/d\eta$, becomes numerically equal to the current true stress, S. This is a general statement, universally true, and quite independent of the strain hardening properties of the material. It holds for compression, where no maximum load occurs, as well as for tension.

Differentiation of the exponential strain hardening function (equation 5 of the text) shows that

$$\frac{dS}{d\eta} = \frac{S_\infty - S}{\eta_c} \qquad (ii)$$

Combining this with equation (*i*), and using the suffix *m* to denote the critical condition, it follows that the critical true stress, S_m is given by

$$S_m = \frac{S_\infty}{1 + \eta_c} \qquad (iii)$$

The corresponding critical strain, η_m, can be found by putting this value of the critical stress into the exponential function.

In general terms, the work done per unit volume, W, is

$$W = \int_\circ^\eta S \, d\eta \qquad (iv)$$

which, in the light of the exponential function, becomes

$$W = S_\infty \eta - (S - S_\circ)\eta_c \qquad (v)$$

The critical work, W_m, is obtained by using the critical stress and critical strain in this equation.

The rate of work hardening, as distinct from the rate of strain hardening, is measured by the differential coefficient dS/dW, which, from equations (*iv*) and (*ii*), is found to be

$$\frac{dS}{dW} = \frac{1}{S} \cdot \frac{dS}{d\eta} \qquad (vi)$$

$$= \frac{1}{\eta_c}\left(\frac{S_\infty}{S} - 1\right) \qquad (vii)$$

Equation (*vi*) is a general statement, universally true and independent of the strain hardening properties of the material, while equation (*vii*) interprets it in terms of the exponential strain hardening function.

At low stresses, in the neighbourhood of zero, the rate of strain hardening, $dS/d\eta$, is always high. Consequently it follows from equation (*vi*) that the rate of work hardening, dS/dW, is invariably infinite at zero stress. Moreover, because of the equality of $dS/d\eta$ and S at the critical point, the rate of work hardening is always unity at the critical point. This is a far more fundamental property of the critical point than the fact that it coincides with the maximum load in tension.

The *specific work* per unit volume, W_s, required to reduce the rate of work hardening from infinity to unity is thus the result of integrating equation (*iv*) between the limits of $S = 0$ and $S = S_m$, or between the corresponding strains of $\eta = -\eta_c$ and $\eta = \eta_m$. By suitable manipulation it can be shown to be

$$W_s = S_\infty \eta_c \left\{ \log_e \left(1 + \frac{1}{\eta_c}\right) - \frac{1}{1 + \eta_c} \right\} \qquad (viii)$$

Thus the specific work depends on the two major strain hardening constants alone. Being independent of the threshold stress and latent strain, it remains unaffected by any moderate degree of deformation (less than η_m), which may have been applied to the metal, either intentionally or inadvertently. This fact, coupled with the fundamental nature of specific work, commends it as a basis of comparison. For annealed materials it is only a little greater than the critical work, but for strain hardened materials (up to $\eta = \eta_m$) it remains constant, whereas the critical work diminishes progressively.

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No. 833 NEKRASOV: "Soviet Blast-furnace Production." *Stal*, 1957, Nov., 965–968. (£2 10s. 0d.).

Jamaican Iron and Copper Finds

HARVEY ALUMINIUM of America have leased over 600 square miles from the Jamaica Government and have now a team in the field engaged in prospecting, according to a report in the current issue of Barclays Bank D.C.O.'s *Overseas Review*. The *Review* also says that at Mavis Bank, about 18 miles from Kingston, the capital, preliminary surface stripping continues to bear out International Metals, Ltd., preliminary estimates of deposits of high grade iron ore and copper ore in commercial quantities.

Progress in the Joining of Aluminium

By N. T. Burgess, A.I.M.
(*Aluminium Development Association*)

Considerable progress has been made in the welding of aluminium and its alloys, and recent achievements include the joining of 6-in. thick plate and, at the other extreme, of thin foil. An account is given of recent developments in welding, brazing and soldering.

THE number and variety of aluminium weldments are increasing all the time, and those who have worked on the improvement of techniques and equipment for welding aluminium will find just reward therein, particularly as many structures now welded were at first thought unweldable. There are no practical limits to size or thickness of aluminium that can be welded: for example, plate for nuclear energy applications has been welded up to 6 in. thick, and at the other extreme the Americans have announced an ultrasonic welding method for joining thin aluminium foil. This article describes some notable welded structures and indicates the progress made in various fields.

Welding Equipment and Techniques

The British Welding Research Association has made a very full study of the arc characteristics of the self-adjusting arc welding process, in conjunction with the Electrical Research Association, and it has been shown that there is a limiting range of operating conditions outside which certain weld defects arise. With low currents and wire speeds (the "sub-threshold" range), metal is transferred in intermittent globules: in the "defective" range, associated with high rates of wire feed and current in excess of 350 amp., serious defects occur in the weld known as "puckering," and in extreme cases more serious internal voids known as "tunnelling" can occur. These defects are connected with gross oxide film entrapment resulting from turbulence.

Fig. 1.—B.W.R.A. welding head for high current welding incorporating secondary argon flow.

As a result of this work, a special welding head has been developed by the B.W.R.A. that incorporates a secondary flow of argon, so that oxidation and blackening of the molten weld pool is prevented by a wide argon coverage (Fig. 1). Currents considerably in excess of those normally usable can be applied without risk of puckering and tunnelling. In this way 1 in. thick aluminium plate has been welded in two passes, one from each side, with an edge preparation of 30° included angle only.

In recent years, much attention has been paid to the type of power source that is best suited to the self-adjusting-arc welding process, and it is believed that certain existing types of power units which have so-called "drooping" characteristics have considerable disadvantages in many applications. The power source is such that as the operating current is lowered so does the voltage change, giving an unstable arc, burn-back and other troubles. Much work has been done by both the equipment manufacturers and private investigators, and a useful paper on this subject has been published by Young.[1] It is now quite clear that for the majority of applications a constant potential power source, i.e. one having stable "flat" characteristics, is best suited to the needs of aluminium welding, and has none of the inherent disadvantages of the conventional unit.

The General Electric Company of America have gone a stage further in developing a power source with a rising characteristic, which is claimed to give even better welding conditions. This equipment, known as Fillerarc, has several variations, including devices not yet available in this country, particularly with respect to wire feed drive. In most British equipment, the wire feed rolls are located near the wire spool at some distance from the welding gun: the rolls have therefore to push the wire through a gun. On the Fillerarc equipment, the drive is actually located in the gun and the wire is pulled through, enabling the passage of much finer gauges of wire. This permits the welding of thinner gauges of aluminium sheet than hitherto, and it is possible to weld $\frac{1}{16}$ in. thick material at great speeds. British equipment is still normally able to weld down to $\frac{3}{16}$ in. only, although by careful manipulation and suitable current settings, satisfactory weld beads on material of $\frac{1}{8}$ in. thickness can be obtained. It should be a considerable advantage to be able to weld material thinner than this, that is in the range currently welded by argon-tungsten-arc methods. At the Commonwealth Welding Conference, it was noted that techniques have been so perfected

Fig. 2.—Tungsten-arc cutting of aluminium plate

that one can now place a weld bead on a plate by inert-gas metal-arc welding without the welding heat affecting the reverse side of that plate. The significance of this is that heat treated, or work hardened materials may not necessarily be softened by the welding process, and this might have considerable value in structural welding, where one often has to design to the annealed strength of the alloy.

The Electrical Research Association, working on the arc characteristics of tungsten-arc welding processes, has developed the surge injection principle, which is a method of striking and maintaining a good arc, as an alternative to superimposed high frequency. This has now been incorporated into the equipment manufactured by two major companies and is considered to be a very worthwhile development. Safer, cleaner and easier welding are noted advantages.

A claim has been made by the Germans that oxygen can be incorporated with advantage into the argon shield. This statement was made in a film that has recently been received in this country on the welding of a large aluminium storage tank. It is noted that 1% of oxygen is used to improve the quality of welds and to avoid preheating. The veracity of this statement is doubted by authorities in this country, and it is presumed that the benefit of oxygen is simply due to its combination with any hydrogen in the molten pool to lower the porosity level. This seems doubtful, and it is felt that the increased risk of oxide film formation and subsequent entrapment does not justify its use.

A new type of high-frequency welding process applied to a tube making machine, known as the Thermatool process, has been developed by the New Rochelle Tool Corporation, of New York. A 25 or 50 kW. power unit is used to provide current at a frequency of 450,000 c./s. to the welding head. As applied to the welding of tubes, it is claimed that the process is 25% faster than other methods, and the quality of the welding is excellent. The principle of operation is that the current takes a path of lower inductance along the V of the tube edges as these are brought together. This path carries nearly all the current, the path of higher inductance being around the back of the tube. Due to the high current frequency, only the metal along the abutting surface edges is heated, and this area reaches weld temperature before the roll pressure is applied. Under pressure, molten metal is squeezed out and metal in the plastic state is brought into intimate contact to form a sound weld. Speeds up to 225 ft./min. are possible on 1 in. o.d. tube.

Arc Cutting

The tungsten-arc cutting of aluminium has at last become a reality, and was shown publicly at the 1957 Engineering and Marine Exhibition.[2] The basic principles are the same as those for argon-arc welding, but moisture-free nitrogen is used as the shielding gas, and a specially constricted, high velocity arc gives a clean cut. For plate thicker than ½ in., a mixture of nitrogen and hydrogen is used. Aluminium up to 2 in. thick may be cut by this method, typical operating conditions for ½ in. thickness being: arc voltage—66 V.; D.C. current—360 A.; nitrogen flow—60 cu. ft./hr.; speed of cut—360 ft./hr. (72 in./min.).

The method is therefore fast and economical, and offers considerable scope for the large aluminium fabricator, although the initial cost of suitable power sources is likely to limit the scope of the process. Water cooled shielding nozzles are required, and a special arc initiation device is fitted. (Fig. 2)

Metallurgy

The aluminium-magnesium alloys have assumed the title of "readily weldable" materials and find widespread use in all applications. In plate form, the alloy NP 5/6 gives high efficiency (commonly 90%) when welded, and finds increasing application in general engineering. Many of the problems associated with the welding of the aluminium—2¼% magnesium alloy have been overcome with the use of a filler rod containing 3½% or 5% of magnesium.

The introduction of the H30 alloy specification, requiring a compulsory manganese content to the familiar Al-Mg-Si alloy, provides a structural material that is weldable. Choice of filler alloy is, however, important if sound welds and good properties are to be achieved.

Work continues on techniques for the satisfactory welding of high strength aluminium alloys of the Al-Cu-Mg and Al-Zn-Mg-Cu types, and certain filler rods have been proposed and patented.[6] Apart from the problem of finding a filler rod that has a high crack resistance, the deposit must have reasonably good strength and ductility, both in the "as-welded" condition and after re-heat-treatment. Rods containing silicon have therefore been used as a basis for experiments, and one of the most satisfactory compositions for H14–H15 alloy contains 3½% silicon, 7% copper, 0·6% magnesium and 0·5% titanium.

Although a satisfactory rod having the requirements outlined above would find immediate use, there are nevertheless very few applications in which these alloys are welded. They are not general purpose materials and are unsuitable for most engineering applications. However, an account appeared recently[7] describing the welding and post-weld heat treatment of a pressure vessel

made in D.T.D. 687A, an Al-Zn-Mg-Cu-alloy. Weld strengths of 34 tons/sq. in. were required, and by careful selection of filler rods and appropriate heat treatment they were obtained. Strong, reliable welds can therefore be attained in these alloys, but whether the attention given to this particular pressure vessel project is warranted in other fields is doubtful.

More promising, perhaps, is the use of Al-Zn-Mg age hardening alloys by some of the Continental countries. In Germany and Switzerland, it is claimed that an alloy containing 4% zinc and 1% magnesium can be readily welded, using either parent metal filler or a silicon alloy rod. After welding, the weld and the heat-affected zone age-harden naturally over a period of three months to provide a tensile strength of up to 25 tons/sq. in. The alloy is claimed to have good corrosion resistance, and has been used for lorry bodies in Germany and for bridge structures in Switzerland.

British Standards

B.S. 1126, on the gas welding of aluminium, has been revised and enlarged to include details of filler rod selection, post-weld working, etc. B.S.1453, on "Filler Rods for Gas Welding," has also been revised, and aluminium filler rods are now dealt with in detail under Section G.

With the inert-gas arc processes now coming into common usage, the need for a British Standard on argon arc welding has become urgent, and it should not now be many months before this is available for general use. A standard for "Filler Rods for Inert-Gas Arc Welding," which has been produced, namely B.S.2901 : 1957, deals usefully with the correct rods for particular applications.

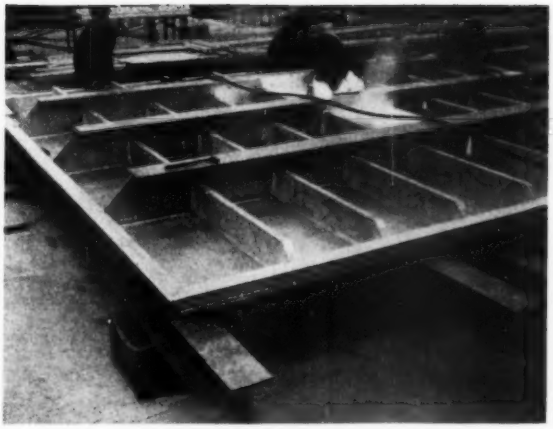
In the absence of standardized procedures, the Aluminium Development Association has published some "General Recommendations for the Testing of Aluminium Fusion Welds and Welders," and these are becoming accepted both in the U.K. and the U.S.A. As the number of fabricators and welding shops working with aluminium increases, so will the demand for standard practices, and the introduction of British Standards can only be regarded as further evidence of the considerable progress made.

Some Notable Welded Structures

The ease with which aluminium can be welded has contributed considerably to the widespread use of the metal for marine structures and for chemical vessels.

Full reports have now appeared of the construction of the first all-aluminium welded superstructure fabricated in a British shipyard. This was for the M.V. *Bergensfjord*, built during 1955/56 at the yard of Swan Hunter & Wigham Richardson, Ltd. The structure incorporates 350 tons of aluminium alloy in the form of plate and section, and 334 miles of welding wire ; it represents one of the biggest structures welded in this country, and results in a weight saving of 500 tons. Twenty-eight welders and twenty welding machines were used on the structure, and full details are given in the literature.[3]

Following directly on the successful use of welded aluminium structures in this way, it is indeed interesting to note that further superstructures are planned for two British shipping lines, the P. & O. and the Orient. It is expected that fabrication of these structures will begin in 1958, at the Northern Ireland shipyard of Harland & Wolff, Ltd., and at the Barrow yard of Vickers-Armstrong, Ltd. The aluminium used in each structure will total approximately 1,000 tons, and will result in considerable weight saving and fuel economies. The shipbuilders have shown foresight in their adoption of welded aluminium, and their welders and welding supervisors are leading in a field that will obviously be of considerable importance in the future. Most shipyards have by now appreciable experience of welding aluminium, and their advice and experience is consequently sought. The symposium organised by five professional institutions and associations in December, 1955, collected together twenty papers on the subject of welding aluminium in shipbuilding, and provided a fine opportunity to discuss controversial matters.



Courtesy Head Wrightson Aluminium, Ltd. and Northern Aluminium Co., Ltd.

Fig. 3.—Welded aluminium deep tank hatch cover.

The opportunity was taken by many interested parties to inspect the S.S. *Sunrip*, which is an ore carrier operating principally between Jamaica and Kitimat and which was built in Canada for Saguenay Terminals, Ltd. This has an all-welded aluminium superstructure and incorporates approximately 120 tons of aluminium, most of which has been left unpainted. The ship has been in continuous service for nearly four years, and the structure generally, and the welds in particular, appear in good condition. Other ore carriers, the S.S. *Sunwalker* built in Japan and S.S. *Sunhenderson*, built in Hamburg, also employ considerable amounts of aluminium in such welded parts as the deckhouse, hatch covers, lifeboats, davits, etc.

All-aluminium welded superstructures are incorporated in U.S. Navy destroyers. Butt welds 20 ft. long are common, and fast deposition rates keep distortion to a minimum. The largest all-welded aluminium structure has also been announced : this is for an elevator unit aboard the U.S. aircraft carrier U.S.S. *Ranger*. It weighs over 1,000 short tons, and was fabricated from plate up to 2 in. thick by the inert-gas metal-arc process.

Also in the marine field, the fabrication of welded aluminium tank hatch covers has been given increased impetus by the availability of a fast welding process. Several such hatch covers measuring about 20 ft. square have been fabricated, commonly in alloy NP5/6, a typical structure being illustrated in Fig. 3. Smaller hatch covers have been made, and the MacGregor type for trawlers was shown at last year's Engineering and Marine Exhibition.

Courtesy A.P.V. Co., Ltd.

Fig. 4.—Welding 6-in. thick aluminium plate by the inert-gas metal-arc process.

Chemical Applications

Another field for which welded aluminium structures have been found to be admirably suited is in the construction of chemical equipment, particularly vessels. Welded aluminium vessels have been made for many years, and a gas welded aluminium tanker made in 1931 is still giving good service. Gas welding has been widely applied in this work, and still proves quite suitable. However, the advent of argon arc and self-adjusting arc welding techniques has meant that such items can be made speedily and economically, and new projects are being contemplated all the time.

It has been found that rocket fuels, particularly high test peroxide (H.T.P.), can only be contained satisfactorily in vessels of 99·8% purity aluminium. The standard of welding has to be very high for this use, and initially isolated troubles were experienced with tungsten inclusions in the welds. Such inclusions can arise in the weld both by unsatisfactory manipulation on the part of the operator, and by breakdown of the tungsten electrode when operating at too high a current or for too long a period. The use of electrodes activated by thoria has largely overcome this trouble, and it is interesting to note that these can be produced more cheaply than pure tungsten. Electrode pick-up does not occur with the consumable electrode process, although troubles have been experienced with deposition of copper in the weld as a result of fusion of the copper contact tube.

Welded vessels for high test peroxide are now well established, and their reliability is a tribute to aluminium welding progress. Recently, a vessel for this purpose has been made from $1\frac{3}{4}$ in. thick NP5/6 plate and pressure tested to 1,200 lb./sq. in.

Now that designers can give more consideration to welded structures in aluminium, much greater use has been made of plate, for example in road tankers and transporters. The carriage of materials in bulk has proved to be an economical system in these days of costly packaging and transport, and for this reason many aluminium bulk transporters are made which carry cement, lime, sugar, etc. These are generally fabricated from alloy NP5/6 in $\frac{3}{16}$ in. or $\frac{1}{4}$ in. thick plate, using the inert-gas metal-arc process. They are pressure tested and the welds are found to be most efficient.

Atomic energy applications are of special interest, since it has been found that aluminium behaves well in this field. Aluminium, because of its low neutron capture cross section, has been used for canning fuels, but it is welded vessels and other equipment that have proved the reliability of aluminium welding techniques (Fig. 4). Several experimental reactor tanks have been made using tungsten-arc and inert-gas metal-arc, and the standard of welding has been very high. Lancaster[4] has reported on some of this work, and notes that although attention must be paid to metallurgical factors and heat flow, it is now possible to fabricate aluminium structures to close dimensional tolerances and to acceptably high welding standards.

On the aluminium tank for the DIDO research reactor made from $\frac{1}{2}$ in. thick PIA-$\frac{1}{2}$H plate, the double-operator argon-arc technique was used with great success, although when welding thicknesses greater than $\frac{1}{2}$ in. the inert-gas metal-arc process gives consistently better quality welds.

Ancillary equipment for nuclear reactors is commonly fabricated from aluminium, and an interesting account of such work was given at the Autumn meeting of the Institute of Welding.[5] Heat exchange equipment and dummy fuel elements have been described, and are typical of such applications.

In recent years, aluminium has been used in the gas and oil industries, and welded aluminium piping is becoming common in many plants. Special joint preparations for aluminium piping have been developed, and in America, where the application is still further developed, the Reynolds Metal Co. have built a special automatic pipe welding machine, which, with the inert-gas metal-arc makes 18 circumferential butt welds per hour, *in situ*. Some of the largest welded aluminium structures are being fabricated for the oil industry, and off shore drilling rigs for Lake Maracaibo have been made from an undisclosed alloy (probably N5/6 type) by semi-automatic inert-gas metal-arc techniques (Fig. 5). It is worthwhile noting the tendency to weld thick-wall aluminium pipes with the initial pass made by the tungsten-arc process, the remaining passes being by the inert metal-arc process.

The chemical industry of Great Britain has always been in the forefront with welded aluminium vessels, and current items of interest are heat exchangers and piping for tonnage oxygen plant. Likewise, the carriage and storage of liquified gases in bulk has been shown to be an economical procedure, and aluminium structures have been proposed to a large extent in this field. The metal has excellent properties at low temperatures, and sea-going tankers in aluminium alloy N5/6 are planned. However, the first stage in this programme will be the erection of large liquid methane storage tanks, some 50 ft. in diameter and 50 ft. high. The alloy will be NP5/6 and these will be the largest storage vessels built on site, to-date.

In the transport field, welded aluminium bodies for tipping lorries are well established, as are platform lorries. In dairying, the welded aluminium milk churn has proved itself to be a successful development; although careful attention to the welding speed and current have been necessary with what is generally considered to be a rather difficult alloy to weld (H30).

Brazing and Soldering

Brazing is still widely applied, particularly for batch

production items such as evaporators, and a salt bath for aluminium brazing has been installed at the works of a radiator manufacturer. The Aluminium Development Association Information Bulletin on "Brazing Aluminium," published in 1957, provides full technical data on the subject. The conventional brazed components are radiators, heat exchangers and wave guides, and there have been no startling developments in this field. The Americans, however, have announced a proprietary compound under the name of Handibraze, which combines the fluxing and filling operation. The compound is brushed, or placed into the joint, and the assembly put into a furnace or salt bath. It is believed that the compound simply combines flux and brazing metal, but preliminary trials in this country indicate that it works well.

New ideas for the soldering of aluminium continue to garnish the Patent Office lists, and the reader of technical journals must be wary lest he be misled into believing that, in the age of the Sputnik, all such technical difficulties are overcome. Many developments have improved the art, but the quest for a low temperature, corrosion resistant solder for aluminium continues. In the meantime, corrosion resistant joints can be obtained by hard soldering, using a zinc-based solder, and low temperature joints can be made with the common tin-zinc alloys, using a flux or one of the recently developed soldering brushes. The situation is conveniently summed up in a new Information Bulletin on "Soldering Aluminium" recently published.[8]

Courtesy Reynolds Metals Co., Ltd.

Fig. 5.—Oil drilling rig fabricated from pipe and plate by inert-gas metal-arc welding.

Technical Meetings

The past year has been one in which greater prominence than ever has been given to the techniques for the welding of aluminium. The seventh and last of the B.W.R.A. Summer Schools included lectures and discussion on the welding of aluminium in both light and heavy engineering industries. The Institute of Welding has now taken over this important educational work, and it is significant that the new School of Welding Technology included a week's course on Welded Design and Construction in Light Alloys in its first syllabus.

If it were needed, further evidence of the continued and growing interest in the welding of aluminium arises from the Commonwealth Welding Conference held in London and Saltburn in June of 1957. Some sixty papers were presented and discussed, and of these eleven dealt exclusively with the welding of aluminium, while many others discussed this subject in line with other developments. Many interesting points emerged from the Conference, and the full proceedings will provide a useful addition to the library shelves, particularly as the welding of aluminium with respect to various industries, e.g. road transport, chemical vessels, etc., is fully covered.

Immediately following this successful Conference, the International Institute of Welding held its Annual Assembly in Essen, Germany, and again it was interesting to note the inclusion of three papers on aluminium. The British contribution was a paper by Dr. W. I. Pumphrey and Dr. E. G. West on the Metallurgy of Welding Aluminium and its Alloys.[8]

This brief survey has indicated the ways in which aluminium welding technology has progressed and only the coming years will show the importance of this in terms of larger and more complicated structures that are already envisaged.

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Carbide Extrusion Dies

For some years, experiments have been made into the use of tungsten carbide for hot extrusion dies, and several firms have developed a technique whereby they can utilise dies in this material economically. A report recently issued by Protolite, Ltd., concerns the extrusion of a magnesium-silicon-aluminium alloy at at emperature of 500° C. There are two distinct types of die in operation, a 0·387 in. diameter single bore and a 0·390 in. double bore die. The former has done 1,540 billets, and both dies look as if they will do a lot more before the bores wear oversize. This is remarkable performance when it is considered that a normal steel die has a life of 120 billets on the average and at the best has never exceeded 500 billets. One further advantage is gained by the use of tungsten carbide dies, namely increased extruding speed: this has now been stepped up from 60 to 85 ft./min.

New and Revised Standards

WROUGHT ALUMINIUM FOR ELECTRICAL PURPOSES—DRAWN STRIP.
(B.S.2897 : 1957.) Price 4s.
WROUGHT ALUMINIUM AND ALUMINIUM ALLOYS FOR ELECTRICAL PURPOSES—BARS AND SECTIONS
(B.S.2898 : 1957.) Price 5s.

THESE two new British Standards for wrought aluminium and aluminium alloys for electrical purposes form a series with B.S.2627 " Wrought Aluminium for Electrical Purposes. Wire." The demand for them arose from the increasing use of aluminium for electrical conductors of many types.

B.S.2897 deals with drawn strip used for electrical conductors such as windings for rotating electrical machines or transformers, its requirements being suitable for controlling the quality of electrical-purity aluminium strip supplied for general electrical components made to other British Standards. B.S.2898 deals with bars and sections used for electrical conductors such as busbars, and components of switchgear, its requirements being suitable for controlling the quality of electrical-purity aluminium and aluminium alloy supplied for components made to other British Standards, e.g. B.S.159 " Busbars and Busbar Connections in Air, Oil or Compound."

Because of the rapid developments in the application of aluminium for electrical purposes, arrangements have been made for both these standards to be reviewed in about two years' time. Users of B.S.2897 and B.S.2898 are invited to submit to the British Standards Institution any comments and suggestions on which to base the intended revisions.

CADMIUM ANODES AND CADMIUM OXIDE FOR ELECTROPLATING
(B.S. 2868 : 1957) PRICE 3s.

THIS is the latest addition to the series of standards dealing with electroplating (there are nine others in the series).

Part 1 specifies requirements for the chemical composition of cadmium anodes ; but since it is impossible to determine the impurities in cadmium by chemical method, it is required that a spectrographic method be used. Part 2 specifies chemical composition, loss on ignition, and insoluble material and impurities. Here again, spectrographic methods are laid down for the determination of impurities. The standard recommends that, wherever possible, British Standard laboratory glassware should be used when carrying out the tests.

WIRE BRUSHES
(B.S. 2939 : 1958). PRICE 7s. 6d.

HAND and rotary brushes filled with wire are the subject of this new British Standard, which has been prepared by representatives of the wire brush industry, local authorities and hospitals, Government Departments and the Services. The new specification deals with wire scratch brushes, casting brushes, platers' brushes, scrubs, brooms, and rotary cup brushes, and with a wide range of types for the removal of carbon, scale, paint, etc., from metal surfaces (when mounted on portable pneumatic or electric tools and on stationary grinders).

Many of these brushes were originally dealt with in War Office Specification No. TG. 26, and while the British Standard may thus be regarded as superseding TG. 26 it includes, at the request of the manufacturers, many brushes not previously standardized. With certain limitations, the Committee has framed the requirements in terms which will include hand-drawn and machine-made brushes, in order that the standard can be applicable to the whole output of the wire brush industry. The specifications are likely to prove helpful to purchasing authorities and will enable manufacturers to simplify their production.

GLOSSARY OF TERMS RELATING TO POWDERS
(B.S. 2955 : 1958). PRICE 4s. 6d.

THIS new British Standard defines a number of selected terms applicable to metallic and non-metallic powders. Following a definition of powder (arbitrarily decided upon as " discrete particles of dry material of size less than 1,000 microns maximum dimension ") are sections containing terms associated with : types of powder, particle size, particle shape, powder properties, and powder processing. The two appendices to the standard deal with " Mean diameters of particulate systems " and " Calculations of Stoke's diameter " respectively.

Copies of these standards may be obtained from the British Standards Institution, Sales Branch, 2 Park Street London, W.1.

B.O. Dounreay Contract

THE use of argon to provide an inert atmosphere plays an essential part in the United Kingdom Atomic Energy Authority's project at Dounreay. The gas employed for this purpose must reach exceptional standards of purity, since even the smallest contamination by air will render it useless. In order to avoid unnecessary waste of this valuable gas, the Authority has awarded a contract to British Oxygen Engineering, Ltd., for the installation of a special plant which will remove all traces of air from the gas and return it to its original state of high purity.

Basically, the process is one of injecting hydrogen into the impure gas to act as a reducing agent by removing oxygen in the form of water, and of subsequent low temperature distillation for nitrogen removal. Refrigeration for the process is provided by the use of liquid oxygen, and the contract includes the provision of two small liquid oxygen plants and a liquid oxygen storage vessel.

Iron Ore from Brazil

THE M.A. Hanna Company of Cleveland, Ohio, through its subsidiary, Hanna Coal and Ore Corporation, has acquired together with Leo Model, of New York, and Associates, the controlling interest in the 125-year-old British Company, St. John D'El Rey Mining Co., Ltd., which for many years has operated the largest gold mine in Brazil, and also has what is believed to be the largest single holding of high-grade iron ore reserves in the country. The deposits of Brazil have long been thought to be the largest high-grade iron ore reserves in the world, estimated at hundreds of millions of tons of ore, containing 60–70% iron.

The Company is to explore fully the iron ore reserves, and to make detailed studies of the transportation of iron ore and dock facilities. If a commercial development proves feasible, it is believed the ore could be marketed competitively in the United States, Great Britain, and countries in Western Europe.

Accelerated High Temperature Oxidation due to Vanadium Pentoxide

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Gas turbines running on residual oils from the distillation of crude oils would be an economic proposition in many marine and land-based applications, but trouble is experienced in the case of oils from several sources in that such oils are rich in a corrosive ash, which clogs up the turbine and attacks metal components operating at high temperatures. This phenomenon has been attributed to the vanadium pentoxide in the ash, either alone or in combination with other substances, and in this article the author discusses the problem in the light of the results of a number of investigations.

A FEW years ago the popular press brewed up a storm in a tea-cup about a Finnish ship carrying "jet fuel" to Communist China. Difficulties were put in the way of the ship and it turned round somewhere in the Indian Ocean and sailed home again. The Press stories managed to convey the impression that some precious fluid with unique properties was being carried to the Chinese air force, whose powerful jets were only waiting to fill their tanks before taking off. The more sober organs of the Press allowed it to be known that the cargo of that solitary Finnish tanker was paraffin, which conjures up somewhat more humble and domestic images. The point of the story is not merely the journalistic skill with which news stories are slanted by the judicious use of words, but the very important fact that the development of the jet engine and gas turbine has provided a new and revolutionary type of power unit which can make use of less volatile, less highly refined, and cheaper fuels.

The fuel consumption of jets, turbo-jets, and turbo-props is rather high, and the economy achieved by the use of cheaper fuels is, of course, always welcome, but the main criterion in the selection of power units is the performance of the aircraft. This is especially true of military aircraft, where costs are met by the taxpayer, but even in the field of civil air transport, where commercial competition plays an important part, fuel costs represent a significant but not a predominant factor. The situation is completely different in the field of marine and land-based units, where the gas turbine is in direct competition with the steam turbine which can run on a variety of fuels, and where capital and operating costs are the main criterion. In many marine applications, and in the field of power generation, gas turbines running on residual oils from the distillation process are an economic proposition, but the use of more expensive fuel would shift the balance in favour of the steam turbine or some other prime mover.

Unfortunately, the residual oils from many sources are rich in a highly corrosive ash, which clogs up the turbine and attacks metal components operating at high temperatures, leading to loss of power and gradual disappearance of the blades. The use of closed-cycle turbines, where the blades are driven by clean hot air (or conceivably another gas) does not solve the problem; the ash build-up and corrosive attack are merely transferred to the heat exchanger or "air boiler," so that heat transfer is slowed down and the operating temperature of the turbine falls, until ultimately the tubes are punctured and the cycle ceases to be "closed."

Oil Ash and its Constituents

Complete combustion of a fuel oil yields nitrogen, carbon dioxide, water vapour, and traces of sulphur dioxide. In practice, the fuel is always burnt with excess air, so that the products of combustion will contain sufficient free oxygen to render the sulphur dioxide relatively harmless.[1] Cheap "residual" oils contain a considerable amount of ash and, as has already been pointed out, this is the cause of all the trouble. The distillation process concentrates all the solid matter present in the original crude in the "residues." The more efficient the distillation, the higher the yield of petrol, and the smaller will be the bulk of residual oil in which the ash is concentrated.[2]

As one would expect, the amount of ash and its composition vary considerably; the literature gives the impression that the variation is primarily between oil from different deposits, but the scatter of analyses of oil samples from the same source is not usually quoted, and it seems likely that, apart from genuine variations in the composition of the crude oil, there must be considerable fluctuation in the degree of contamination by solid "dirt" in normal oil-well practice, and probably some variations in sampling techniques. In addition, some constituents begin to volatilise at relatively low temperatures, and the amount lost in analysis will depend on the time and temperature of "burning off," which may vary in different laboratories. As an example of the variations which may be encountered, the ash contents and the contents of vanadium pentoxide in the ash from samples of residual oil taken at weekly intervals at four American refineries[2] are collected in Table I. It is evident that the main variation is geographical, but there is considerable fluctuation in vanadium pentoxide contents in samples from the same source.

There is hardly a metal in the periodic table which has not been detected in some oil ash at some time, but the bulk of the ash usually consists of various compounds of the alkali metals, iron, nickel, vanadium and sulphur. Many constituents of the ash are present as solid particles suspended in the oil. This does not apply to vanadium, however, which occurs as organic compounds;[2] in combustion, the vanadium is oxidised and is found in the ash in the form of vanadium pentoxide. Vanadium is generally associated with asphalt-base

TABLE I.—VARIATION OF TOTAL ASH AND VANADIUM PENTOMIDE CONTENTS OF WEEKLY SAMPLES OF RESIDUAL OILS PRODUCED BY FOUR REFINERIES.[2]

Refinery		Sample									
		1	2	3	4	5	6	7	8	9	10
A	% Ash					0·049	0·046	0·054	0·055	12·34	
	% V_2O_5 in ash					12·3	10·5	22·6	13·8	7·9	
B	% Ash		0·033	0·043	0·041	0·041	0·051	0·039	0·040	0·034	
	% V_2O_5 in ash		38·4	59·8	62·8	43·1	26·2	39·0	36·6	35·9	
C	% Ash	0·027	0·029	0·026	0·035	0·026	0·022	0·021	0·028	0·024	0·024
	% V_2O_5 in ash	13·3	12·3	18·9	9·2	15·0	13·6	12·0	12·6	14·5	13·0
D	% Ash		0·051	0·038	0·034	0·086	0·082	0·067	0·048*	0·059	
	% V_2O_5 in ash		1·6	4·9	0·8	1·5	0·9	1·9	2·6	1·8	

* In the original this figure is 0·0048, but this is presumably a misprint.

crudes.[3] This suggests that vanadium was present in the plants which decayed to form the petroleums and bitumens.

Analyses of oil ash from numerous sources, derived from a moderately comprehensive survey of the literature, are presented in Table II. Most authors report ash compositions in terms of the oxides found, or those most likely to be formed by combustion with plenty of excess air present, but one series of results is quoted in terms of the metallic elements, and this is relegated to the bottom of the table. The ash compositions quoted show considerable variations, but it is apparent that in this, as in other fields, the United States are very fortunate in having substantial supplies of raw materials of a type which is not likely to give serious trouble. Only one type of crude oil (Oklahoma[3]), contains more than 20% V_2O_5. Even the South American and Middle East crudes quoted by American authors[8] have lower vanadium contents than many of the "industrial fuel oils," presumably from the same areas, for which analyses are quoted by European authors.[9, 11, 12] The reason for this discrepancy is not to be sought in any deliberate tendency of the oil suppliers to send high-vanadium crude to Europe and low-vanadium to America, but rather in a "built-in bias" in the table; the American papers aim at a broad survey of the supply position, while the European analyses are extracted from papers dealing with laboratory experiments or trial runs on turbines, using fuel oils deliberately selected to assess the incidence of oil ash corrosion.

Apart from the analyses in Table II, a few isolated vanadium contents have been culled from the literature. The oil ash of a Persian crude was reported to contain 5·03% V_2O_5,[4] that from a Venezuelan crude 45% V_2O_5,[5] while another South American oil was alleged to yield an ash containing up to 80% V_2O_5;[7] quite high vanadium contents have also been found in coal ash.[7]

Although it appears that America is fairly well off for residual fuel oils of relatively low vanadium content, the problem of oil ash corrosion is considered sufficiently serious there to warrant a research effort which, although substantial only by European rather than American standards, has yielded interesting results.[7, 27, 28, 31] The reason for this concern is to be found in the high transport costs of low-vanadium crudes to areas where high-vanadium crude is indigenous,[3] and to some extent in the fact that it would be over-optimistic to assert that any oil ash containing more than 1% vanadium pentoxide is really safe from the point of view of corrosion. Nevertheless, it is reasonable to conclude that the problem is on the whole more serious and more acute in Europe than in America, partly because low-vanadium crudes are not as easily available, and partly because the development of the gas turbine for power generation is of considerable economic importance in Europe.

THE EFFECTS OF OIL ASH

Fouling and Scaling in Gas Turbines

Oil ash corrosion of metal components exposed to high temperatures is a generally known and reluctantly

TABLE II.—TYPICAL COMPOSITIONS OF OIL ASHES (%)

(a) In terms of oxides

Source	Ash Content	SiO_2	Combined Oxides	Fe_2O_3	MgO	CaO	Na_2O	K_2O	P_2O_5	MnO	V_2O_5	SO_3	NiO	Li_2O	Chloride	Ref.
California Crude		38·79	22·38	(7·71)	1·81	8·68	9·51	—	—	0·302	(5·07)	15·02	4·35	0·144	—	3
Wyoming Crude		40·31	51·48	(44·59)	1·19	3·45	2·58	—	(0·06)	0·198	trace	1·44	0·33	0·163	—	
Midcontinent Crude		31·68	31·80	(15·81)	4·18	12·62	6·90	—	—	0·437	trace	10·80	0·533	—	—	
Texas Panhandle Crude		1·55	10·32	(7·74)	2·49	5·26	30·80	1·03	—	0·33	(1·43)	42·11	1·52	—	4·64	
Pennsylvania Crude		0·75	97·52	(97·37)	0·239	0·68	0·129	—	—	0·213	—	0·875	—	—	—	
Kansas Crude		9·99	19·55	(14·69)	1·27	4·79	23·60	0·922	—	0·038	(0·438)	36·41	0·571	—	0·10	
Oklahoma Crude											(22·14)		5·89	—	—	
British Industrial Fuel Oil ..	0·047	10·00	4·00	—	—	5·00	15·00	—	—	—	25·00	30·00	6·0	—	—	10
" " " " ..	0·044	—	5·5	—	—	5·00	3·7*	—	—	—	62·00	1·85	4·7	—	—	11
Swiss Industrial Fuel Oil ..	0·07	—	—	—	—	—	8·7	—	—	—	66·6	12·8	—	—	—	12
" " " " ..	0·047	—	0·6	—	—	—	11·5	—	—	—	67·2	7·8	6·0	—	—	9
" " " " ..	0·035	—	2·6	—	—	—	26·5	—	—	—	39·0	14·3	9·6	—	—	
" " " " ..	0·048	—	—	—	—	—	13·76	—	—	—	65·8	—	—	—	—	
" " " " ..	0·038	—	—	—	—	—	2·36	—	—	—	82·3	—	—	—	—	
" " " " ..	0·04	—	—	—	—	—	6·2	—	—	—	81·6	—	—	—	—	

* Na

(b) In terms of elements

Source	Si	Fe	Al	Ca	Na	Cr	Pb	CO_3	V	S	Ni	H_2	Ref.
United States 1	10·0	9·5	10·0	7·5	5·0	0·5	0·1–1·0	ind	6·0	10·0	5·0	—	8
2	1·0	46·5	1·0	0·1–1·0	1·0	1·5	present	7·5	3·0	8·0	0·5	—	
Venezuelan 1	1·0	6·4	3·0	0·1–1·0	2·0	1·5	present	0·5	44·5	3·0	3·5	0·2	
2	2·0	5·5	3·0	0·1–1·0	1·0	4·0	present	1·5	35·0	0·15	7·5	—	
Middle East	1·0	3·5	2·0	0·1–1·0	15·5	7·0	1–10	0·5	18·0	7·5	3·5	—	

accepted feature of heavy-oil fired boilers in marine or industrial applications; super-heater supports and baffle plates operate at about 800° C., and are usually made very bulky so that they give a sensible service life.[13] The problem has also been encountered in some oil refinery stills.[7] In gas turbines, the remedy of increasing the metal section and allowing the metal to corrode away is not merely expensive, it is downright impossible. The weight of the moving parts has to be kept down to the minimum which will support the stresses; increasing the weight of the blades would increase the centrifugal stresses to a level where they would burst the rotor. In the heat exchanger of the closed cycle turbine, too, excessive tube thickness would slow down heat transfer. Moreover, these alloys are very expensive, and while it does not matter if an occasional support or fastening is thicker than it ought to be, a substantial increase in the gauge of the tubing used in the construction of the air boiler would make the closed circuit turbine uneconomic.

The main problem of oil ash corrosion arises in large marine and industrial gas turbines. In practice, no major breakdowns have been reported, because the

Fig. 1. Turbine blading after 250 hours on gas oil.[6]

severe corrosion of metal parts was detected in experimental turbines, and until the problem is solved no large gas turbine with any stage operating above 650° C. will burn residual fuel oil.

The gas turbine was developed during the war, and until the war was over all efforts were concentrated on aircraft applications. The experience gained with gas turbines in aircraft was utilised in the design of experimental industrial turbines immediately after the war, and the first reports of oil ash corrosion were reported in 1948.[14,c/12]

A detailed account of the effect of the use of residual fuel oil in an experimental gas turbine is given by Bucher.[6,13] The installation was operated as an open-cycle turbine. The rotor blades in the first two stages were divided into groups, each being made of a different material: Rex.337A, G18B, Staybrite F.C.B. and cold worked F.C.B., Nimonic 80 and Nimonic 80A, were used. The turbine was run on gas oil for 700 hours with little trouble and only slight oxidation of the blades (Fig. 1). The fuel was then changed to a residual oil containing 0·046% ash and 0·0139% vanadium.

"After the first 10 hours it was noted that the net

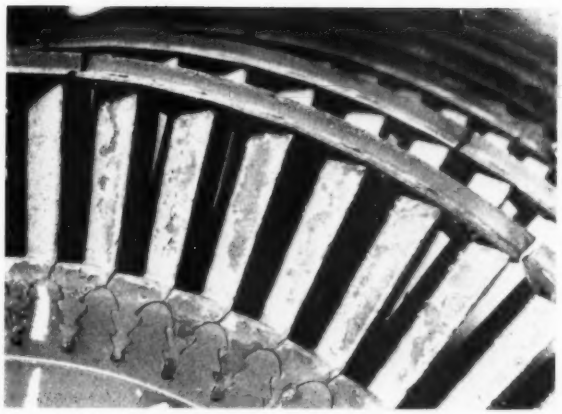


Fig. 2. Turbine blading after 300 hours run on B.P. Britoleum.[6]

output of the plant began to fall off, but it was decided to keep running to see whether this deterioration continued. After the first 200 hours 48% of the original output was lost Measurements showed that part of this loss was due to compressor fouling and part to throttling of the turbine." Measures were taken to restore the compressor efficiency and these " restored the output somewhat, but on starting up again and continuing the test to the 300-hour mark the output continued to fall. The turbine had not been opened up during the whole of this test and it was now decided to shut down and examine the blading.

" Fig. 2 shows the rotor blading after this run, and it may be seen that there is a considerable amount of scaling, and that an amount of solids had collected under the shrouding. As can be seen from Fig. 3, the main trouble was on the nozzle plate, where heavy deposits had collected. It should perhaps be noted at this stage that throughout the test on heavy oil the pressure at the compressor outlet had shown a tendency to rise, and in fact there was a difference of over 2 lb. between the beginning and the end of the test. This indicated that the turbine was in fact getting smaller. This conclusion was supported by the mass flow readings, which dropped from 9·5 lb./sec. at the beginning of the test to 8·7 lb./sec. after 300 hrs.

Fig. 3. Nozzle blading.[6]

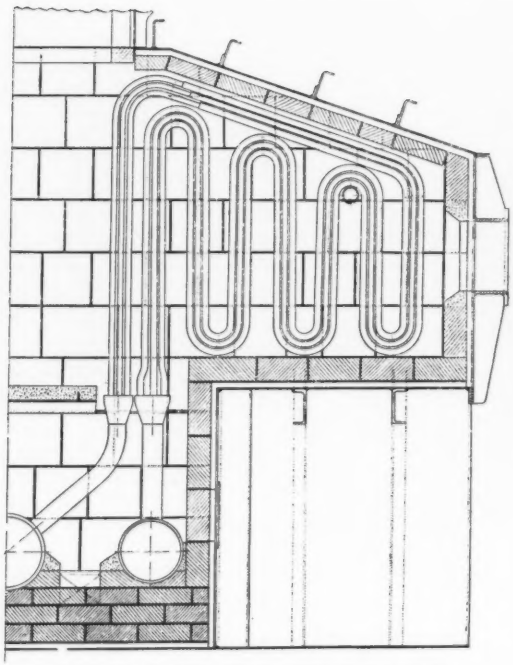


Fig. 4.—Half section through air heater.[6]

" An examination of the deposits in the turbine showed the following composition :—

Nickel oxide	13·7%
Chromium oxide	18·6%
Iron oxide	48·4%
Silica	2·4%
Sulphate	3·4%
Vanadium pentoxide	8·3%
Copper oxide, lime, magnesia ..	traces

" The rotor blades, except those made from materials having a high nickel content, showed distinct signs of scaling, and a more careful examination showed that all the blades, again with the exception of those having a high nickel content, had been attacked by the vanadium pentoxide. This vanadium attack was only noticeable in the first two stages, that is, roughly, where the temperature was above 600° C. From these tests it would appear that some considerable difficulties will be experienced in running open-cycle gas turbines on anything other than distillates, unless a method of cleaning the blading without shutting down the plant can be developed."[6]

The plant was subsequently converted to a closed cycle by substituting an air heater (Figs. 4 and 5) for the combustion chamber. The initial run of 76 hours with gas oil showed no serious corrosion of the turbine or the air heater. The unit was then operated for 7 hours with a residual oil, and the cover was removed from the air heater tubing " but no noticeable deposits were found on the tubes and everything appeared to be quite in order. An examination of the combustion chamber did not disclose any carbon deposits, with the exception of a slight build-up on the face of the sprayer ; the unit was therefore closed again and the testing continued. After the initial tests with B.P. Britoleum (residual oil), that is after the closed circuit had completed some 80 hours running, the compressor was opened up for examination, but was found to be in a very clean state with no

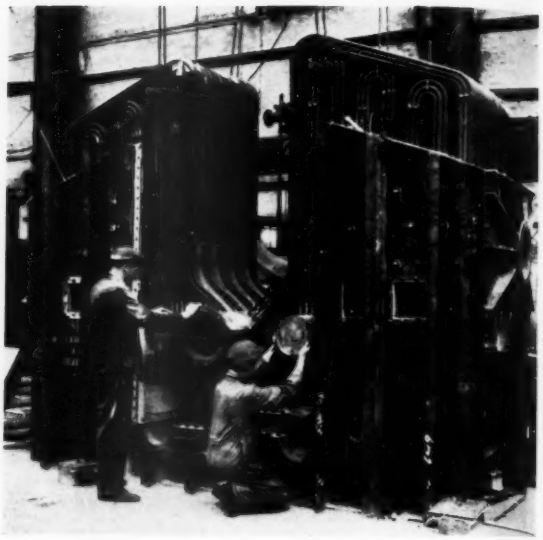


Fig. 5.—Tube nest during assembly.[6]

deposits on the blading, except for a little fine dust which was found to be mostly ferrous oxide and can be assumed to have come from the new piping used on the circuit. No measureable difference in compressor performance was noted during this running period. A careful examination of the temperatures and pressures in other parts of the circuit showed that there had been no alteration to the turbine, the regenerator, or the cooler, in fact all the circuit conditions after test were exactly as they had been initially."[6] The plant was then run for a further 200 hours. " During this run a small quantity of carbon tetrachloride was injected into the combustion chamber, with the idea that volatile chlorides of vanadium would be formed which would pass through the air heater. Unfortunately, at the high temperatures in the combustion chamber, these chlorides appeared to break down into vanadium pentoxide and chlorine, with the result that the attack on the tubes was more serious than it would have been if there had been no carbon tetrachloride present. As it was, the corrosion on the tubes at the end of the test period was very severe."[6] It is reasonable to suppose that, even in the absence of the carbon tetrachloride addition, severe corrosion was to be expected.

" Fig. 6*a* shows one of the more heavily corroded tubes after a 200 hour test at 800° C. ; the attack was so serious that pinholing occurred in parts of the tubing. The material used was a complex nickel-chromium-cobalt austenitic steel—one of the best of the present-day creep-resisting alloys. Alloys of the 12/18 nickel-chromium niobium-stabilized materials were also used in the air heater ; the attack on these was considerably less (see Fig. 6*b*), although it was so severe that the tube would have a life of only 2,000–3,000 hours in an industrial plant."[13]

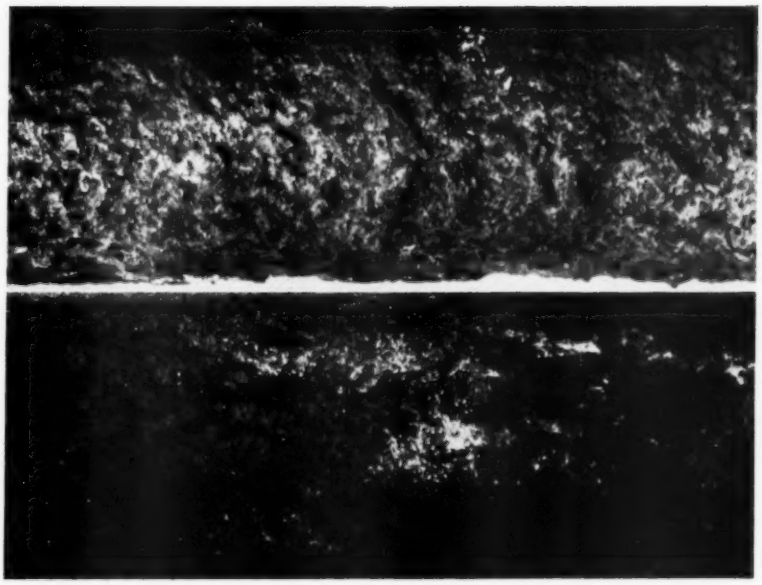
In another experimental turbine,[11, 15] considerable fouling but no corrosion was observed after 3,000 hours running on a variety of fuels, including vanadium-bearing residual oils, at temperatures between 565 and 590° C., with a short period at 650° C. It would appear that the lower operating temperature accounts for the absence of

oil ash corrosion in this case. Unfortunately, the efficiency of the gas turbine is much higher at high gas temperatures, and a rigid limitation to 650° C. is not really acceptable.

Some gas turbines are in operation for power generation in Switzerland, and in one of these residual fuel oils were burnt for experimental periods. "Fouling was found particularly on the blading of the output turbine, made of G18B steel, and hence the steel has also suffered excessive corrosion. Very heavy fouling was recorded with an oil with 1·9% of sulphur and an ash content of 0·047%.

"The analysis of the ash was as follows :—

V_2O_5	67·2%
NiO	6·0%
Fe_2O_3	0·6%
Na_2O	11·5%
CaO	3·2%
SO_3	7·8%
Undetermined components	0·7%



(a)

(b)

Fig. 6.—Air heater tubes after a 200 hour test at 800° C. : (a) complex nickel-chromium-cobalt austenitic steel tube ; (b) 12/18 nickel-chromium-niobium steel tube.[13]

"The analysis of the fouling deposit on the turbine blades varied according to the temperature of the relevant blade part. The corresponding values were :—

	First Stage 650° C.	Last Stage 550° C.
C, %	0·21	0·69
SiO_2%	7·96	5·97
Fe_2O_3%	14·20	1·60
CaO%	4·21	4·48
MgO%	Trace	Trace
Cr_2O_3%	3·80	1·01
V_2O_5%	48·61	50·85
NiO%	6·01	4·23
SO_3%	7·34	21·21
Na_2O, K_2O%	5·62	9·08
H_2O%	0·34	0·52
Melting point	780° C.	550° C.
Temperature	650° C.	550° C.
Colour	Dirty brown	Yellow

"The ash deposits at lower temperatures contained much more sulphate and alkali than those at higher temperatures ; further, the melting point of the ash deposited at high temperature is about 100° C. higher than of that deposited at lower temperatures.

"Using another fuel with 1·85% of sulphur and an ash content of 0·035%, comprising :—

V_2O_5	39·0%
NiO	9·6%
Fe_2O_3	2·6%
Na_2O	26·5%
CaO	2·4%
SO_3	14·3%
Undetermined Components	5·6%

the fouling deposits were considerably less. At various stages of the turbine the composition of the deposits was as follows :—

Stage	Temp. ° C.	V_2O_5 %	Fe_2O_3 %	SO_3 %	Na_2O %
1	650	40·9	23·1	12·1	5·1
3	620	36·4	27·1	12·7	5·8
5	590	34·5	24·7	14·7	7·8
9	560	34·9	2·2	26·1	12·5

"The fouling is therefore essentially determined by the vanadium content of the oil, that is, the vanadium pentoxide, which, in a pasty state, sticks to the blades. The effect of other components of the ash has not yet been determined."[9]

Corrosion in Laboratory Tests

So much for practical experience with experimental turbines. There are limits to the variables which can be studied in a turbine, and investigations of the mechanism of corrosion, the effect of different mixtures, and the resistance of different alloys, are of course carried out in the laboratory.

The first step in a laboratory investigation is an attempt to reproduce the effect observed in practice. In the case of oil ash corrosion, as in many others, far more effects have been produced experimentally than were ever detected in practice. On the other hand, one effect observed in trial runs in turbines was only reproduced in the laboratory much later.[26] Some investigations are more painstaking than others in imitating practical conditions. Often they start off with elaborately controlled oil-firing systems and finish by putting a small heap of vanadium pentoxide on a piece of alloy sheet and heating the specimens in air ;[12] synthetic atmospheres, based on burnt town's gas, are sometimes used,[10] and the corrosive reagents may be added a little bit at a time, in imitation of the continuous deposition and build-up of ash in the turbine.[16] In some instances the specimens were partially immersed in a crucible containing molten vanadium pentoxide,[21, 7] or exposed to a slow stream of pure oxygen and dipped into molten vanadium pentoxide every quarter of an hour.[40] Although it is useful to know the effect of these variables, it appears to be generally agreed that the important factors are time, temperature, total quantity of reagents, mixture of reagents, composition of the alloy, and the presence of sulphur-bearing gases ; all but the last can be

Fig. 7.—Typical effects produced on cylinders of 16/25/6 chromium-nickel-molybdenum alloy by heating in still oxidising atmospheres.[20]

studied conveniently in simple laboratory air-muffles.

A judicious mixture of the constituents present in oil ash will increase the rate of oxidation of all materials suitable for use in the gas turbine, but the effect on different alloys varies both in extent and in kind. The sharpest distinction is between austenitic steels and nickel-based alloys; the former suffer 'catastrophic' oxidation in the presence of vanadium pentoxide, while the latter show merely a substantial increase in the rate of oxidation, but are rendered much more susceptible to the detrimental influence of sulphur in the oil ash or the atmosphere.

Catastrophic oxidation was first observed in austenitic steels containing molybdenum;[20] on heating in still air at temperatures above about 815° C., they rapidly form a spongy scale with a characteristic 'pie-crust' appearance illustrated in Fig. 7. The phenomenon has been attributed to the accumulation of molybdic oxide (MoO_3) vapour on the metal surface. The spongy scale formed as a result of catastrophic oxidation does not show distinct layers consisting of different oxides, but a single, highly porous layer. Inert markers applied to the surface of the unoxidised specimen are retained on the outer surface of the scale layer,[20, 7] indicating that scaling proceeds entirely by penetration of oxygen through the fissures and pores of the scale and oxidation at the metal/scale interface. The surface below the spongy scale is roughened, but there is no intergranular attack or loss of ductility.[20, 7] Leslie and Fontana, who were the first to study the phenomenon in detail,[20] found that vanadium pentoxide and, in a lesser degree, a variety of other oxides, accentuated and facilitated the catastrophic oxidation of an austenitic steel containing 6% molybdenum, and that both molybdic oxide and vanadium pentoxide increased the rate of oxidation of a variety of other alloys free from molydenum. They are a little ambiguous in discussing the nature of the scales formed, however. Thus, they state that the distinctive types of oxide formed by 16/25/6 (16% Cr, 25% Ni, 6% Mo) steel and similar alloys were not found in any of the alloys tested in this series of molybdenum-free steels, although these included a variety of stainless steels of relatively low alloy content, and even pure iron. On the other hand, they also observe that "PbO, V_2O_5, WO_3 and Bi_2O_3 have been found to produce the same effects as MoO_3 when present as gases on the metal surface" and they attribute the characteristic scale on the 16/25/6 alloy to the effect of molybdic oxide. Moreover, in a later paper,[21] Fontana describes the work of one of his research students, R. M. Berry, who heated specimens half immersed in molten vanadium pentoxide and found severe attack on the slag line, and states quite unequivocally that vanadium pentoxide produces catastrophic oxidation similar to molybdic oxide. Brasunas and Grant[27] also regard the effects of vanadium pentoxide and molybdic oxide as identical. I have seen specimens of 18/8 free from all suspicion of molybdenum, which formed a characteristic 'pie-crust' scale as a result of

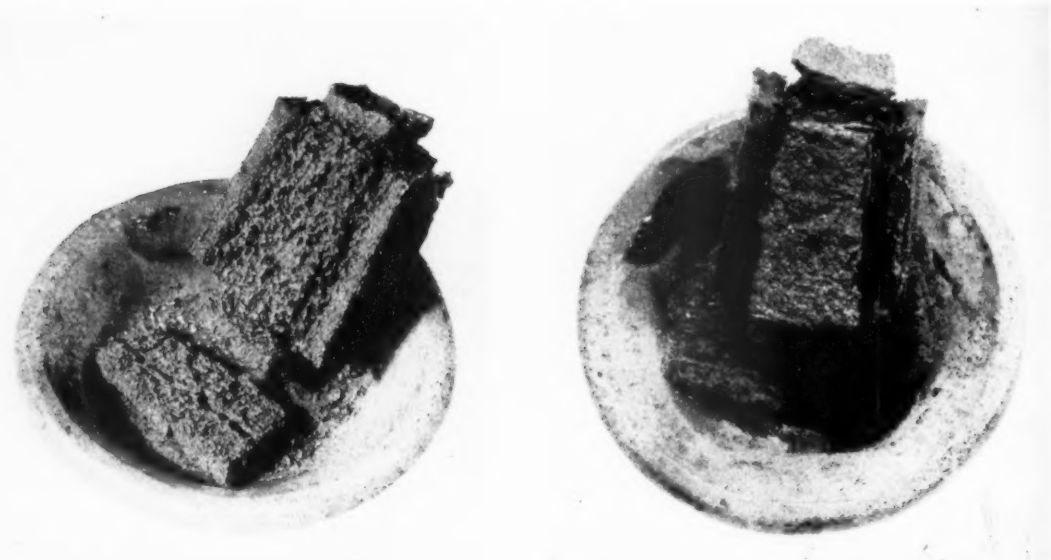


Fig. 8.—Effect of painting 18/8 chromium-nickel steel with vanadium pentoxide and heating for 24 hours at 1,000 C.

extremely rapid oxidation in contact with vanadium pentoxide, and therefore incline to the view that the effect of vanadium pentoxide on austenitic steels can reasonably be described as 'catastrophic oxidation' analogous to that observed in molybdenum-bearing alloys. (Fig. 8.)

(a)—The Effect of Time

The formation of a protective scale on heat-resistant alloys separates the metal and the atmosphere. Oxidation takes place only by counter-current diffusion of metal and oxide ions across the scale, or by diffusion of metal ions to the surfaces; the thicker the scale, the longer the diffusion path becomes, and oxidation is therefore parabolic with time. In catastrophic oxidation, where the fissures in the scale allow free access of the atmosphere to the metal, oxidation depends on the rate of reaction at the surface of the metal, which is independent of the thickness of the scale formed, and is therefore linear with time. An experimental curve[12] is shown in Fig. 9; it refers to specimens of 18/12 and 25/20 Cr-Ni steels which had 10 mg. of vanadium pentoxide or oil ash placed on them and were heated in an air muffle at 850° C. for periods from 6 hours to 480 hours. Owing to rapid penetration of the sheet samples the scatter is rather high, but it appears that oxidation increases rapidly at the beginning and then proceeds more or less linearly. A slight tendency for the curves to flatten out towards the end might be explained by dilution of the vanadium pentoxide with the oxides formed on the sheet.

In another investigation,[7] samples of different alloys were half immersed in 10–15 g. of ash, and the decrease in thickness was compared after 168 hours and 504 hours at 725° C. "The indicated increase in the rates of attack were more than linear, either above the ash, or below the ash or both." The supply of corrosive reagents was no doubt adequate to keep a linear reaction going, but continuous acceleration of the process is difficult to explain, and in view of the fact that the evidence is based on two experimental points only, no attempt at an explanation need be made.

The results of experiments on sheet specimens of various austenitic steels, exposed at 850° C. for 100 hours and 250 hours with about 5 and 10 mg. V_2O_5, are plotted in Fig. 10.[16] Curves drawn through two experimental points and the origin are apt to assume any shape that lends itself to interpretation. Moreover, it is obvious that at least one of the experimental results for the 18/8 steel with 3% Mo is wildly inconsistent. Nevertheless, results tend to point to the interpretation indicated in Fig. 10, that there is a sharp initial increase in the weight loss with time, which levels off during the first 100 hours. These results are in direct contradiction to those of the Swiss workers,[12] shown in Fig. 9; the experimental conditions in the two investigations were similar, and I know of no facts which might help to resolve the contradiction, but in later sections I shall quote some independent experimental evidence tending to support the results of Betteridge, Sachs and Lewis,[16] and put forward some considerations which might explain the wide differences in experimental results.

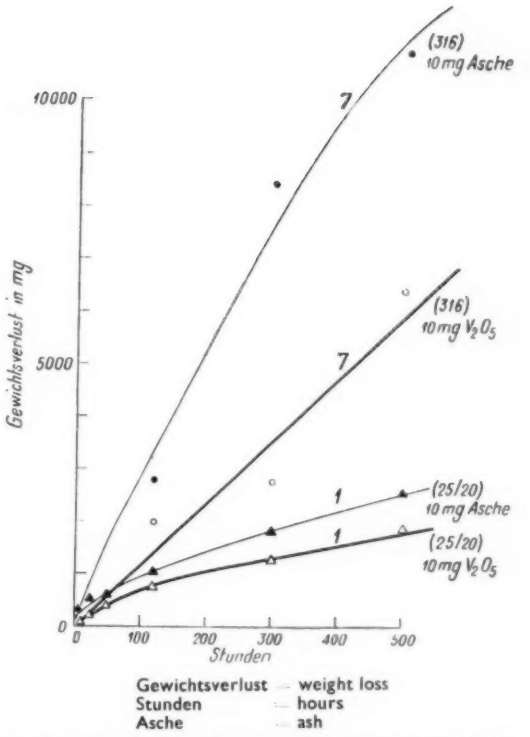


Fig. 9. The effect of time on the attack at 850° C.[12]

The most satisfactory method of determining rates of oxidation is continuous weighing during oxidation. Burns[24] has carried out such determinations on specimens of G 18B with small additions of vanadium pentoxide, and has detected an inflection before the onset of the linear curve. The position of the inflection and the rates of oxidation varied with different amounts of vanadium pentoxide, and this effect will be discussed in more detail below.

(b) The Effect of Temperature

The influence of temperature is fairly straight-

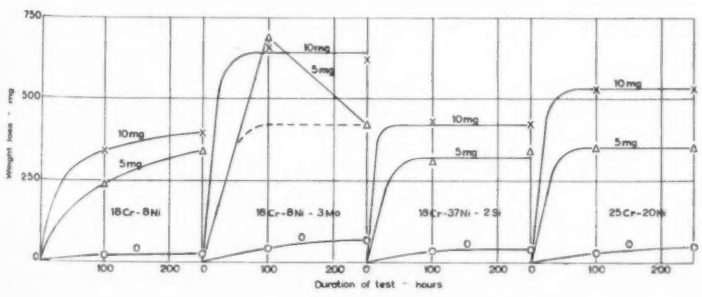


Fig. 10.—Attack on sheet specimens of various austenitic steels exposed to vanadium pentoxide at 850° C. for 100 and 250 hours.

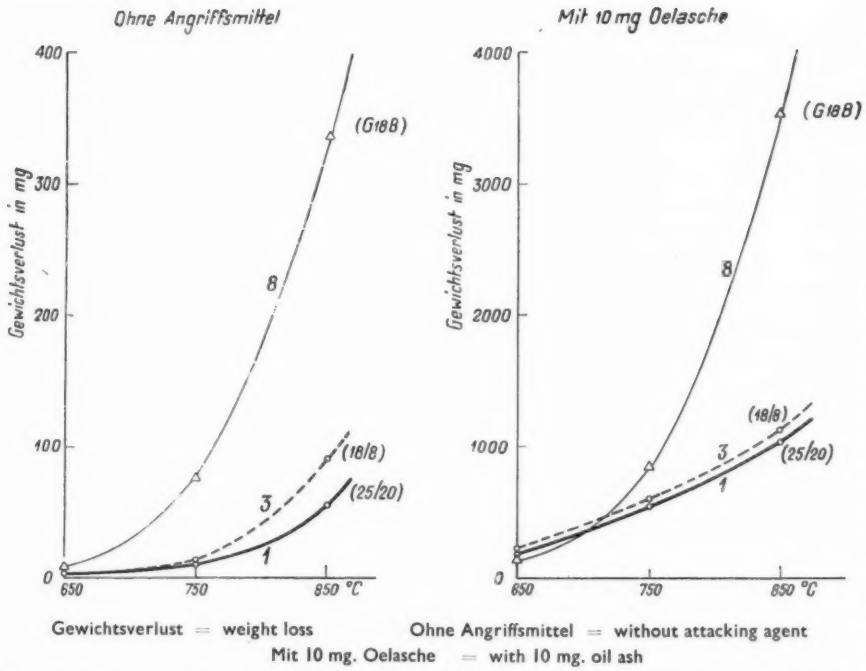


Fig. 11.—Comparison of the weight-losses after heating various austenitic steels for 120 hours, without oil ash and with 10 mg. oil ash.[12]

forward, and there is no serious disagreement among different investigators. There is a threshold temperature below which no significant increase in corrosion rate can be detected. The actual value of this temperature differs slightly for pure vanadium pentoxide, oil ash, and various synthetic mixtures, but it invariably coincides with the melting point of the corrosive agents;[12, 72, 5] this is also true for catastrophic oxidation initiated by molybdic oxide.[17] Above the threshold value, increase in temperature leads to a sharp rise in the rate of oxidation. This behaviour does not differ in principle from normal oxidation, except that the weight losses involved are much higher. Typical curves[12] for oxidation at different temperatures, with and without contaminant, are shown in Fig. 11. The graphs of weight loss against temperature are very similar, particularly for G 18B steel, but they are drawn on different scales, and in the presence of 10 mg. oil ash the weight losses in 120 hours at any temperature are ten times higher than those for similar tests without the corrodant. It is possible to think of vanadium pentoxide as producing at relatively low temperatures effects which would occur in its absence at fairly high temperatures. It is known, for example, that when the scale on nickel-chromium steels fuses at 1,150°–1,200° C., there is a sharp rise in the rate of oxidation.[10]

Some accelerated attack at temperatures below the melting point of the corrodants has been detected in copper-based alloys, particularly aluminium bronze, which is of some interest in gas turbine heat exchanger applications.[7, 25]

(c) The Effect of the Quantity of Contaminant

In considering the effect of the quantity of vanadium pentoxide, it is worth bearing in mind the amounts likely to be deposited in a turbine. Davis[24] estimates that "for a typical engine in which the blading comprises 60% of the wetted area, and assuming an ash content of 0·04% with 75% active constituents, the potential rate of delivery of ash is 3 mg./cm.²/hr. Assuming a fall-out of 10%, the amount of oil ash deposited is 0·3 mg./cm.²/hr., or 7·2 mg./cm.²/day." The severity of the catastrophic oxidation effect increases with the quantity of contaminant. The results of exposures for 120 hours at 650, 750, and 850° C. of small specimens of G 18B sheet, with small heaps of vanadium pentoxide or oil ash piled on them,[12] indicate that 10 mg. of contaminant causes a very substantial weight loss, which is not quite doubled when 20 mg. is used, and increased by a factor of 2 to 3 when the heap of contaminant weighs 50 mg. At a deposition rate of 0·3 mg./cm.²/hr., gradual build-up on one square centimetre would yield 36 mg. in 120 hours; the area covered by the heap of powder is probably of this order of magnitude. Similar results for a variety of alloys exposed for 100 hours and 250 hours at 850° C., with varying amounts of vanadium pentoxide are summarised in Tables II and III, Ref. 16, and plotted in Fig. 12. The general trend of the curves is similar to those obtained by Schläpfer, Amgwerd and Preis,[12] although the actual values of the weight loss are appreciably lower. The fact that the curves for 100-hour and 250-hour tests roughly coincide is due to the levelling-off of the rate of oxidation shown in Fig. 10, which gives the same experimental results plotted against time.

The same paper[16] describes stress-rupture tests on various alloys using small specimens, whose gauge lengths carried coatings of various mixtures of vanadium pentoxide and sodium sulphate. In one series of tests, on straight 18/8 steel, increasingly heavy coatings of pure vanadium pentoxide were employed. Stress rupture tests were carried out at 750° C. and 3 tons/sq. in.:—

Weight of Contaminant (mg.)	Life (hours)	Weight Loss (mg.)
0	94·6	34·4
0·3	70·0	92·8
19·8	35·0	271·2
72·5	26·1	545·9

Here again the effect of small additions is very pronounced, and although an increase in the weight of contaminant gives a further stimulus to oxidation, it is by no means proportional.

Reference has already been made to a study by Burns[24] of the oxidation at 750° C. of G 18B contaminated with vanadium pentoxide, in which the weight

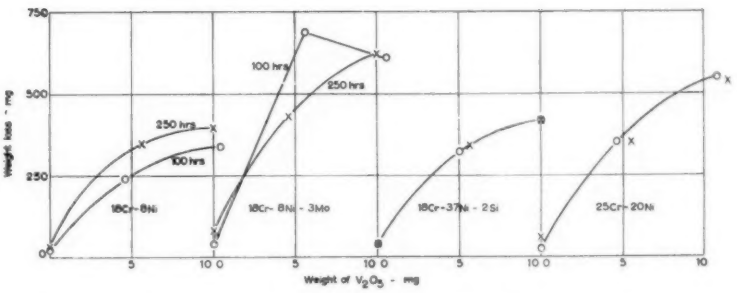


Fig. 12.—Attack on a variety of alloys exposed to varying amounts of vanadium pentoxide at 850° C. for 100 and 250 hours.

gain due to oxidation was recorded continuously. The amount of contamination was extremely heavy, and sharp inflections were observed in the rate of oxidation (Fig. 13). According to Burns, the inflection occurs earlier and at lower weight gains the smaller the amount of contaminant. With 1·2 g. V_2O_5 the inflection occurs after about 120–150 hours at a weight gain of about 160 mg., with 0·6 g. V_2O_5 after about 80 hours at a weight gain of about 80 mg., and with 0·1 g. V_2O_5 the inflection cannot be detected on the graph. The weight gain corresponds to the quantity of oxygen involved in scale formation; in the oxide layers, 48 g. oxygen correspond to 160 g. Fe_2O_3, 152 g. Cr_2O_3, and 71 g. NiO. The scale formed by catastrophic oxidation consists substantially of ferric oxide (Fe_2O_3), nickel oxide (NiO) and chromic oxide (Cr_2O_3) in roughly the proportion in which iron, nickel, and chromium are present in the alloy.[20, 27] In alloy G 18B, a weight gain of 1 g. corresponds to the formation of 3·5 g. scale consisting of 2·6 g. Fe_2O_3, 0·5 g. Cr_2O_3 and 0·4 g. NiO. At the inflection points, therefore, 0·56 g. scale and 1·2 g. V_2O_5, or 0·28 g. scale and 0·6 g. V_2O_5, are present. The approximate composition of the mixture at the inflection would be 68·2% V_2O_5, 23·6% Fe_2O_3, 4·4% Cr_2O_3, and 3·8% NiO. If the inflection always occurs at a a composition of this order, it should take place at a weight loss of 13·3 mg. in the test with 0·1 g. V_2O_5, and would certainly be too close to the origin on the graph in Fig. 13 to be detected.

The melting points of binary mixtures of vanadium pentoxide with other oxides have been determined by Lucas, Weddle and Preece.[23] In the system Fe_2O_3–V_2O_5, a melting point of 750° C. occurs in a mixture containing 76% V_2O_5, 24% Fe_2O_3. It seems reasonable to suggest, therefore, that in the early stages of scaling the contaminant is liquid and oxidation is very rapid indeed, that the formation of other oxides gradually raises the melting point of the mixture of contaminant and oxide until it reaches the test temperature of 750° C., when the mixture becomes solid and an inflection occurs in the rate of oxidation. From a theoretical point of view this explanation is so attractive that it is almost inevitable that experimental evidence should appear which contradicts it. All the alloys used for high temperature application form scales consisting of ferric or chromic oxides, and should form mixtures melting at 750° C. with about 76% V_2O_5; but Burns himself points out that not all alloys show an inflection.[24]

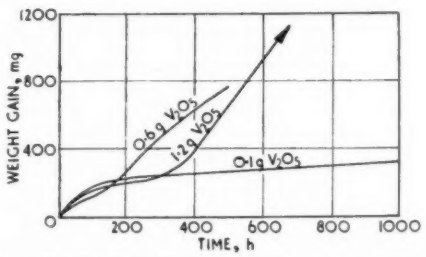


Fig. 13.—Weight-gain/time curves for specimens of G.18B steel heated at 750° C. in contact with vanadium pentoxide.[24]

The curve for 0·1 g. V_2O_5 in Fig. 13 shows a significant slowing-down of oxidation after about 120–150 hours at 750° C., and appears similar to the oxidation curves in Fig. 10. The curves for tests with heavier contamination were not continued long enough to show whether a second levelling-out occurs after the inflection. The inflection also upsets the relation between weight gain and quantity of vanadium pentoxide, but very little extrapolation would be required to indicate that after the inflection has been passed larger quantities of vanadium pentoxide lead to considerably but not proportionally more severe oxidation.

The combined effects of time and increasing amounts of contaminant have been studied by making small additions of the contaminant at regular intervals. Fitzer and Schwab[40] developed a rapid sorting test: specimens were held at 925° C. in a current of oxygen and immersed in molten vanadium pentoxide at 900° C. every quarter of an hour. After an arbitrary number of cycles (in practice after 2, 10, and 20 cycles), the specimens were descaled by quenching in water and immersion in molten caustic soda containing some potassium permanganate. The descaled specimens were weighed to determine the loss of weight due to oxidation; the curves, illustrated in Fig. 14, are substantially linear, and do not show any appreciable effect of the intermediate removal of scale. Under the conditions employed in this test, where the specimens are virtually flushed in molten vanadium pentoxide, the weight of contaminant might be expected to have the predominant effect. To all intents and purposes the curves are linear and do not show the slight levelling-off indicated in Fig. 12

Experiments by Harris, Child and Kerr on a very complex austenitic steel and a cobalt-based alloy (G 18B and G 32) heated up to 1,120 hours in moving air at 850° C. with additions of about 1·5 mg./cm.² of V_2O_5 every 70 hours,[22] also gave a straight-line plot of weight loss against time (Fig. 15). Such a graph could be obtained if the rate of oxidation with the first addition were linear with time and further additions had no further effect, which is highly improbable in view of the results already recorded; or if the curve for the rate of oxidation in the first 70 hours consisted of a steep curve gradually merging into a straight line and the steep rise were reproduced by each addition of vanadium pentoxide, so that the graph really consisted of a series of

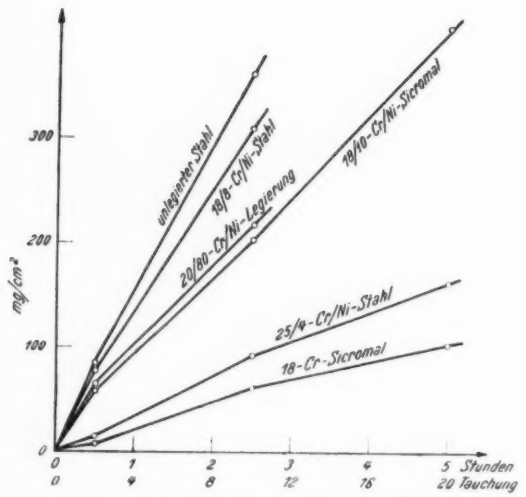


Fig. 14.—Weight loss of specimens heated in oxygen at 925° C. with periodic immersion in molten vanadium pentoxide.[10]

equal steps (Fig. 16). These steps are similar to the graphs of weight loss against time shown in Fig. 10. Altogether, the straight lines in Fig. 15 are not consistent with the observations of Schläpfer, Amgwerd and Preis,[12] that the rate of oxidation is linear and the effect of increasing the quantity of contaminant is a nearly linear increase in weight loss, for in that case each step on the graph corresponding to an increment of vanadium pentoxide would be a straight line of steeper slope than the preceding one (Fig. 16—curve *a*) and the overall curve would become progressively steeper. An alternative explanation for the straight lines in Fig. 15 is that the rate of oxidation depends on the content of vanadium pentoxide in the scale. This would also lead to a graph consisting of uniform steps, but the shape of the step need not resemble that in Fig. 10. However, such an interpretation would imply that the curve of weight loss against time for a single application of vanadium pentoxide at the beginning of the test should show a continuous decrease in the rate of oxidation as the vanadium pentoxide is diluted by the oxide formed. It can be concluded, therefore, that the results of Harris, Child, and Kerr[22] confirm the conclusion outlined in Fig. 10 that the rate of oxidation is initially very steep and levels out after a fairly short time.

It is now possible to summarize the effect of the quantity of contaminant :—

(1) The addition of contaminant causes a spurt in the rate of oxidation.

(2) This spurt is constant in magnitude for a given weight of contaminant, at whatever stage in the progress of oxidation the addition is made.

(3) An increase in weight of contaminant causes an increase in the magnitude of the spurt, but this increase is not quite proportional.

Obviously an upper limit to the effect of increasing weight of vanadium pentoxide is reached when the specimens are immersed in the contaminant. Under those circumstances, it might be expected that dilution of vanadium pentoxide with other materials would reduce the attack. An experiment on these lines was carried out by Sykes and Shirley,[4] who made a mixture of sodium and nickel salts and vanadium pentoxide, corresponding in composition to the analysis of a residual oil ash, and diluted it with a similar mixture free from vanadium pentoxide. Various austenitic steels were immersed in these contaminants, but a significant decrease in oxidation was only detected when the total content of vanadium pentoxide had been lowered to 1%.

It would appear that the influence of the weight of contaminant on the rate and mechanism of high temperature corrosion is worth more detailed experimental investigation. Curves of weight loss against weight of vanadium pentoxide, taken on different alloys and for different periods of exposure, should be very instructive.

(d) The Effect of Other Constituents

Vandium pentoxide on its own causes catastrophic oxidation on some alloys—mostly iron-based—and normal oxidation at an increased rate on others—mainly those based on nickel and cobalt. Together with other compounds which are frequently present in oil ash, the vanadium pentoxide may be responsible for corrosive attack of enhanced severity, particularly on alloys which are less sensitive to pure vanadium pentoxide. This type of attack has two distinguishing features : it is not a general surface attack, but tends to be localised and intergranular, and it is demonstrably stimulated by vanadium pentoxide, because the other contaminant in the absence of vanadium pentoxide does not produce the effect.

An interesting example is quoted by Evans,[7] who exposed Inconel contaminated with a Venezuelan oil ash containing 44·5% V, 3% S, 1·5% Na (Table II*b*), for 168 hours at 732° C. The intergranular attack produced, illustrated in Fig. 17, is ascribed to sulphur penetration. Its probable causes are more fully understood as a result of recent work by Shirley,[26] and will be discussed below.

Intergranular attack by mixtures of vanadium pentoxide has been found in two complex austenitic steels (F.C.B.(T) and G 18B), which suffer accelerated general surface attack in the presence of vanadium pentoxide alone.[25] The grain boundary penetration shown in Figs. 18*a* and 18*b* was produced in 200 hours at 670° C., just above the melting point of the mixture. F.C.B.(T) often contains an intergranular phase after severe attack by contaminants containing vanadium pentoxide. This constituent can be clearly seen at fairly high magnifications after suitable etching treatment, but it does not

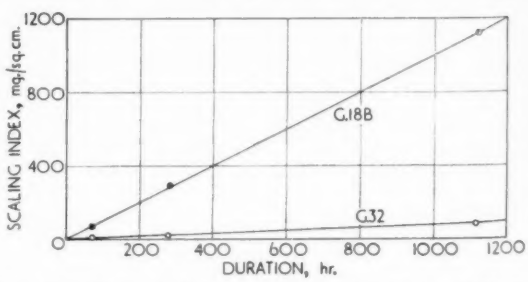


Fig. 15.—Effect of time on the resistance of G 18B and G 32 steels to vanadium pentoxide attack at 850 C. in moving air.[22]

appear to be brittle or to induce selective oxidation, so that it can be considered harmless.[29]

In an attempt to assess the effect of general and intergranular corrosion in a single test, tensile pieces exposed to a residual fuel at 800° C. were broken,[30] and the loss of tensile strength due to exposure was interpreted as decrease in effective cross-section. The penetration into the metal estimated in this way is plotted against penetration figures calculated from the loss of weight after descaling, in Fig. 19. Only one alloy, on the border line between an austenitic steel and a nickel alloy, falls on the theoretical line corresponding to equal penetration values in both tests, e.g. completely uniform surface attack. All other materials tested, both high nickel alloys and austenitic steels on a 25/20 or 18/8 basis, show more severe loss in strength than loss in weight, suggesting intergranular attack. The weight losses of the nickel alloys were negligible, while the austenitic steels suffered severe general corrosion as well as pronounced intergranular attack. The diagram could be interpreted to show that the severest intergranular attack found in an austenitic steel extended as far below the heavily oxidised surface (25/16 steel) as the worst penetration in a nickel alloy extended below the lightly oxidised surface (alloy C). Although such an interpretation neglects the very important influence of surface roughening by the general corrosive attack, it indicates that

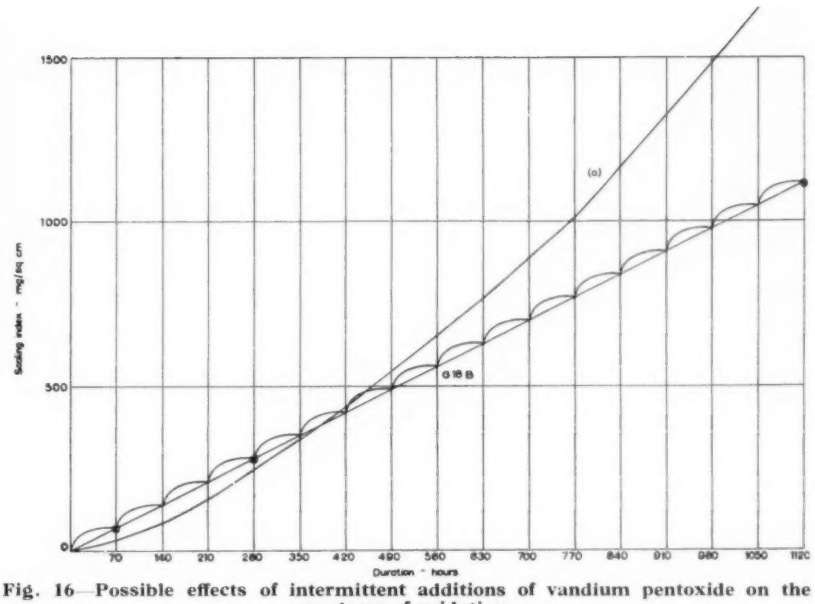


Fig. 16—Possible effects of intermittent additions of vandium pentoxide on the progress of oxidation.

there is some weakening of austenitic steels beyond that attributable to weight loss only; whether this is due to surface roughening or to a grain boundary constituent, the effect on mechanical properties is not as harmless as has been suggested[29] for the intergranular phase in F.C.B.(T), which is not after all so very different in composition (11½% Ni, 17½% Cr, 1¼% Nb).

Mixtures containing vanadium pentoxide may cause both severe general attack and intergranular penetration of iron-based alloys, grain boundary penetration only in nickel-based alloys. Stress rupture tests at 750° C. on bars painted with mixtures of vanadium pentoxide and sodium sulphate[16] indicate that nickel alloys which suffer very little loss in weight must be weakened in some way, suggesting intergranular penetration, even though no microscopic evidence could be detected until the temperature was raised to 1,000° C. In similar tests, austenitic steels had even shorter stress rupture lives; weight losses were extremely heavy and no intergranular attack could be detected. The inference was that there is a general tendency to intergranular penetration, but in many cases general corrosion is so rapid that the scale advances into the metal as rapidly as the intergranular attack, and the latter is overtaken and swamped by the general scaling.

The most important constituents which accompany vanadium pentoxide and enhance its detrimental effect in oil ash are sodium salts, sulphur-bearing gases or salts, and chlorides. These are often in combined form, e.g. sodium sulphate or chloride, but their effects have been studied separately to some extent.

Sodium salts, for instance, have quite a variety of effects. In the absence of other contaminants, sodium carbonate has a corrosive effect on cobalt-based alloys containing molybdenum.[31] In mixtures containing vanadium pentoxide and in residual oil ash, the properties of sodium salts present may influence the rate of attack. Thus, in experiments on 25/20 Ni-Cr austenitic steels,[12] various mixtures of vanadium pentoxide and caustic soda were melted together, and specimens with

Fig. 17.—Photomicrograph showing grain boundary attack on Inconel heated for 168 hours at 1,350 F. (732 C.) in contact with a Venezuelan oil ash. Below ash line—unetched.[7] ×100

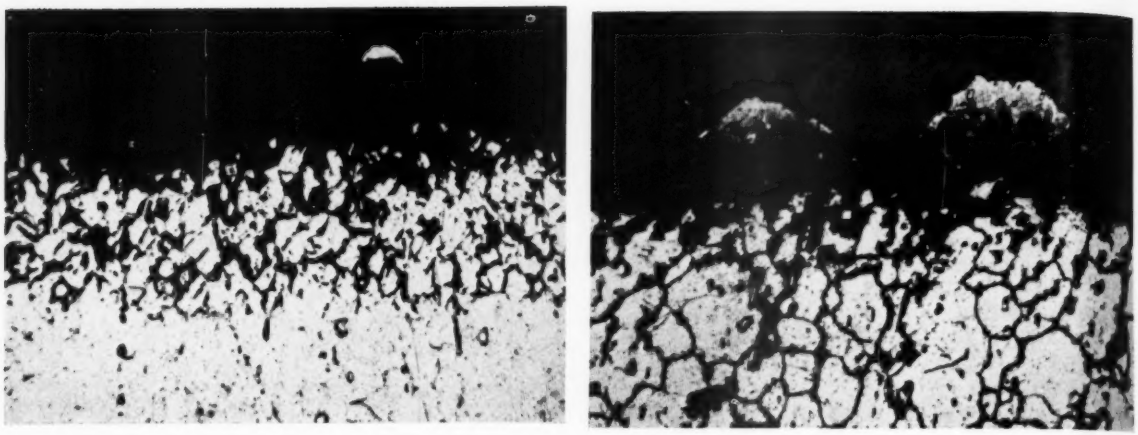
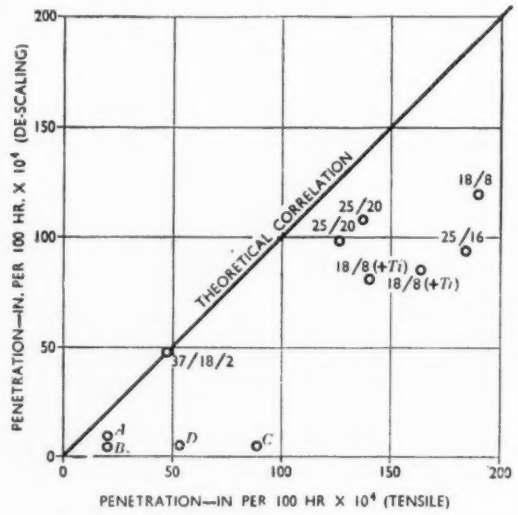


Fig. 18.—Photomicrograph showing corrosion due to exposure for 200 hours at 670° C. to ash composed of vanadium pentoxide and 10% sodium sulphate : (a) F.C.B. (T) steel ; (b) G 18B steel.[25]

10 mg. of this mixture were heated at 850° C. ; when the ratio of alkali to vanadium pentoxide was less than 1, increasing alkali content accelerated the attack, but any further increase beyond that ratio tended to weaken the attack again. This effect is attributed to the formation of vanadyl vanadate and other unstable compounds which may act as oxygen carriers. Similarly, at the National Gas Turbine Establishment,[32] very rapid uniform corrosion was observed with " ashes of moderate sodium/vanadium ratio, particularly those corresponding in composition to the sodium vanadyl vanadates." At high sodium/vanadium ratios, localised attack was found. Tests on austenitic steels and nickel alloys with roasted mixtures of sodium orthovanadate and vanadium pentoxide at 650–800° C. showed the most severe corrosion at an Na_2O/V_2O_5 ratio of 1 : 4 (corresponding to the composition of sodium vanadyl vanadate).[33]



A 78% nickel, 19% chromium, 0·6% silicon.
B 80% nickel, 14% chromium.
C 75% nickel, 20% chromium, 0·6% silicon, 2% titanium.
D 56% nickel, 20% chromium, 19% cobalt.

Fig. 19.—Comparison of corrosion rates on steels and nickel alloys deduced by tensile and weight loss methods.[30]

It has been shown that additions of sodium sulphate lower the melting point and raise the fluidity of vanadium pentoxide,[28] and that the gain in weight of heat-resisting austenitic steels goes through a maximum at about 15% Na_2SO_4 at temperatures of 705° C. and 760° C., where an increase in the fluidity of the melt may play a significant part, but diminishes consistently with increasing content of sodium sulphate at 837° C., where the fluidity of pure vanadium pentoxide is presumably very high. Stress-rupture tests at 750° C. on bars painted with binary mixtures of vanadium pentoxide and sodium sulphate containing 0, 25, 50, 75, and 100% V_2O_5 showed that different heat resistant alloys varied widely in their response to the different mixtures ;[16] austenitic steels generally suffered most severe attack by pure vanadium

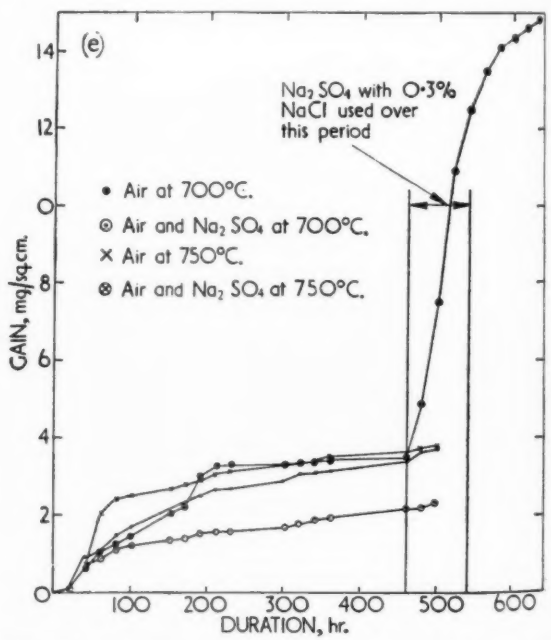


Fig. 20.—Oxidation curves for F.C.B. (T) steel heated at 700 and 750° C. in air, with and without sodium sulphate.[30]

(a)

Fig. 21.—Nimonic 75 after exposure to the products of combustion of Bunker C fuel oil[10]: (a) surface of specimen; (b) section through excrescence on bar. ×100

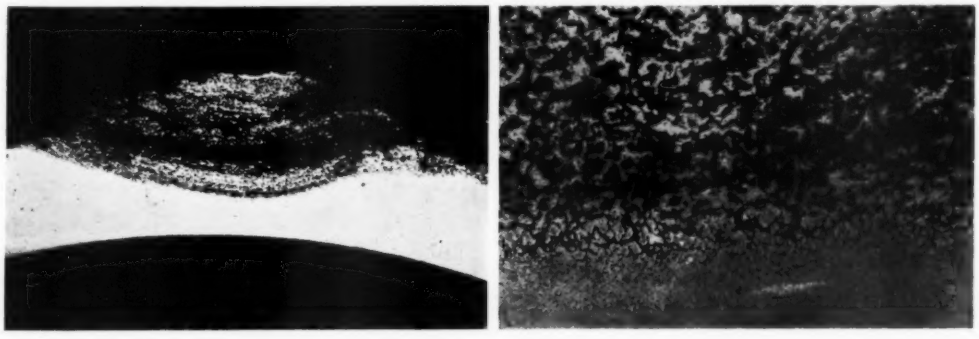
pentoxide, while nickel-based alloys seemed to be more susceptible to the mixtures, particularly 75% V_2O_5, 25% Na_2SO_4. Partial immersion of 18/8 stainless steel sheet specimens in V_2O_5-Na_2SO_4 mixtures at 732° C. revealed no great variation between 10 and 80% V_2O_5; the heaviest attack was found in pure vanadium pentoxide.[7] Frederick and Eden[25] claimed that "preliminary" experiments—which were not described in detail—on F.C.B. (T), G18B and Nimonic 80 showed that a mixture containing 10% Na_2SO_4 gave the most severe attack.

Sodium sulphate may be responsible for severe corrosive effects on its own. These can operate at temperatures considerably below the melting point of the salt (884° C.); although oxidation at 700–750° C. in air is only slightly accelerated by sodium sulphate, the presence of traces of chloride, sulphide compounds in the gas, or incompletely burnt fuel, greatly enhances the effect of sodium sulphate.[10] The effect of as little as 0·3% NaCl is illustrated in Fig. 20 where the addition of pure sodium sulphate results in a parabolic oxidation curve while sodium sulphate contaminated with sodium chloride causes very rapid "linear" scaling.

It has been shown recently[26] that mixtures of sodium sulphate and chloride (and also of the calcium salts) containing 1% or more of sodium chloride may cause intergranular sulphur penetration in heat resistant steels and nickel alloys, leading to the formation of warts on the surface which had previously been attributed to the interaction of vanadium pentoxide and sulphur.[7,16] A typical example of sulphur penetration has already been illustrated in Fig 17, showing an Inconel specimen immersed in oil ash. Similar attack can be seen in Fig. 21 on a specimen of Nimonic 75 exposed to the products of combustion of Bunker C fuel oil, and in Fig. 22 on a Nimonic 80A air heater tube after 1,016 hours at 770° C. in a peat trial. Attempts to reproduce this phenomenon in a laboratory experiment failed because everybody was convinced it was due to vanadium pentoxide in combination with some sulphur compound. However, Shirley[26] succeeded in reproducing this sulphur penetration by small chloride additions; Fig. 23 shows a Nimonic 80A specimen heated 6 hours in air in contact with 99/1 Na_2SO_4—NaCl mixture, and the surface penetration is sufficiently similar to that in Figs. 17, 21 and 22 to warrant the conclusion that they all stem from the same basic cause. Experimental results quoted in Table III show that the attack is particularly severe on Nimonic alloys, and that a mixture containing 10% sodium chloride is most drastic in its effects.

(b)

Warts produced in the presence of vanadium pentoxide often show two zones; the excrescence itself consists of a mixture of oxide and metal impoverished in chromium, and beneath it there is a layer in which the metal has been penetrated by a light phase which has been provisionally identified as Cr_2S_3.[16] Thus, sulphur penetrates into the metal, which loses chromium by the formation of sulphide and is consequently more easily oxidised. In the outer layers, particularly if oxidation is stimulated



(a) (b)

Fig. 22.—Unetched section through Ninomic 80A air heater tube after 1,016 hours at 770° C. in peat trial[26]: (a) ×10; (b) ×100.

TABLE III.—EFFECT OF VARIOUS SALT MIXTURES AT 750° C. IN AIR (6 HOURS)[26]

Test	Contact Mixture	Loss after NaOH Descaling (mg./sq. cm.)			Remarks
		337	F.C.B. (T)	Nim. 80A	
a		1·4	0·9	0·4	
b	100% Na_2SO_4	1·7	0·9	0·7	
c	99% Na_2SO_4, 1% NaCl	11, 12, 4	14, 13, 12	34, 34, 53	Localized "crater" attack on immersed Nimonic.
d	90% Na_2SO_4, 10% NaCl	101, 103, 23	5, 6, 3	295, 452, 497	Very severe local attack on Nimonic with formation of eutectic globules; salt mixture fused.
e		131	5	169	Ditto
f	75% Na_2SO_4, 25% NaCl	99, 10, 111	3, 4, 3	91, 57, 49	Ditto
g	50% Na_2SO_4, 50% NaCl	37, 31	28, 43	16, 19	Fairly uniform attack, even on Nimonic.
h	100% NaCl	16	13	31	Localized "crater" attack on immersed Nimonic.
i	99% K_2SO_4, 1% NaCl	0·8	0·8	0·3	
j	100% $CaSO_4$	32, 44	36, 49	1·0, 1·3	
k	99% $CaSO_4$, 1% NaCl	33	10, 14	1·3, 1·6	
l	99% $CaSO_4$, 1% $CaCl_2$	53, 47	44, 45	5, 4	
m	90% $CaSO_4$, 10% NaCl	77	47	2, 2	Scaling of the steels in these mixtures was usually rather patchy, with flaking on cooling, often leaving the samples practically scale-free on brushing.
n	90/10 $CaSO_4$-Na_2SO_4 with 1% NaCl	8, 19	8, 7	0·6, 0·6	
o	50/50 $CaSO_4$-Na_2SO_4 with 1% NaCl	19	10	1·0	

by the presence of vanadium pentoxide, the sulphide phase is also oxidised.

In austenitic steels (e.g., 337 and F.C.B. (T)) mixtures of sulphates and chlorides produce intergranular corrosion which does not affect the ductility of the material ([26, cf. 29]). Nickel alloys containing a light grey phase also appeared reasonably ductile in a bend test. In tests with mixtures of sodium salts in the absence of vanadium pentoxide, the scale contained some spherical particles consisting of Ni-NiS-NiO eutectic mixtures.[26] This suggests that the excrescences result from the formation of the liquid ternary eutectic, which liquates to the surface in the form of "sweat droplets," leaving the solid chromium sulphide behind in the metal. In most cases, particularly if vanadium pentoxide is also present, the nickel sulphide is oxidised so that in its final condition the wart is solid.

Sulphur penetration of nickel alloys generally occurs only in reducing atmospheres, and the severe attack observed in the presence of sulphate-chlorides mixtures, particularly when the oxidising character of the atmosphere is enhanced by vanadium pentoxide, is perhaps the most puzzling feature of this corrosive effect, the basic mechanism of which remains obscure.

Other solid additions may also cause accentuation. Even additives which inhibit the vanadium pentoxide effect may, in certain concentrations, cause oxidation of increased severity.[25] This work will be quoted in more detail below.

The effects of gaseous impurities have not been studied very intensively. They can generally be predicted from the known behaviour of the corresponding salts. Thus, chlorine is likely to stimulate sulphur penetration if sulphates or sulphur trioxide are present; hydrogen sulphide would cause sulphur penetration in nickel alloys if it could survive without being oxidised: in fact, all sulphur-bearing gases are likely to be converted to sulphur trioxide in the long run, and will probably form sulphates. It is rather curious that sulphur-containing atmospheres, which are often regarded as universally detrimental, may have a beneficial effect in reducing the attack of Na_2O-V_2O_5 mixtures by forming a sulphate in preference to sodium vanadyl vanadate.[32]

Corrosive attack by vanadium pentoxide is often accentuated by other oil ash constituents, but this effect is only observed on alloys which are fairly resistant to pure vanadium pentoxide. Thus, high-nickel alloys of the Nimonic type, which show much less increase in oxidation rate or shortening of life to rupture in the presence of pure vanadium pentoxide than do austenitic steels, may suffer significant attack if the vanadium pentoxide is mixed with sodium sulphate. Conversely, sodium sulphate, which acts as a stimulant to oxidation by vanadium pentoxide of nickel alloys, may act as a diluent which diminishes vanadium pentoxide attack on creep resistant steels.[16]

(to be continued)

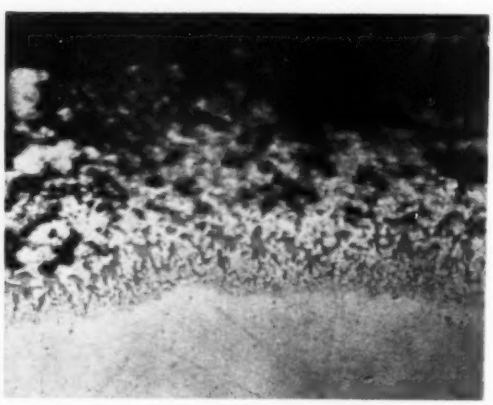


Fig. 23.—Unetched section through Nimonic 80A cylinder tested 6 hours in air at 750° C. in contact with 99·1 sodium sulphate-sodium chloride mixture.[26] × 500

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Correspondence

THE IDENTITY OF THE TAU- AND Y-PHASES FROM AUSTENITIC STEELS

The Editor, METALLURGIA.

Sir,

Brown, Clark and Parker recently described in this journal[1] an inclusion phase isolated from Cr-Ni-Ti steels by electrolytic extraction. The phase seemed well-defined, and a set of values of interplanar spacings (d) was obtained from its X-ray powder diffraction photograph taken with CoK-radiation. Evidence from chemical analysis suggested that the formula of the phase was Ti_2S. This is of interest, since a titanium sulphide of this composition has not been reported in the literature.

Brown *et al.* named the phase *tau* and considered it to be identical with the *Y*-phase reported by Gemmill *et al.*[2] in 8% Cr-Mo-Ti steels. The latter authors did not establish the composition of *Y*, but they believed it to be a polymorph of TiN or a second nitride of titanium. They indexed the lines of *Y* in a composite powder diffraction pattern provisionally on the basis of a hexagonal unit cell, $a = 3{\cdot}206$ Å, $c = 11{\cdot}19$ Å, $c/a = 3{\cdot}49$. The agreement between the observed and calculated values of d was very good (Table I), and the indexing is likely to be correct. It is interesting to note, however, that the diffraction pattern of *tau*, assumed by Brown *et al.* to be identical with *Y*, can be indexed equally satisfactorily on the basis of a tetragonal unit cell, $a = 3{\cdot}92$ Å, $c = 7{\cdot}43$ Å, $c/a = 1{\cdot}901$. Not knowing the quality of the diffraction data for *tau*, no attempt has been made to refine these values further.

When the diffraction pattern of *Y* is indexed tetragonally, the calculated values of d give a much poorer fit than the values based on the hexagonal unit cell, particularly for the high-angle reflections, while for *tau*, with the existing data, the tetragonal indexing leads to a slightly closer agreement of d_o and d_c than the hexagonal indexing. It is possible that *tau* could be indexed hexagonally to give as good a fit as that obtained by Gemmill *et al.* for *Y*, if the d-values for *tau* were of a quality comparable to that reported for *Y*, where the back-reflection technique was used. Possible overlap of some of the high-angle lines of *Y* by lines of another constituent in the composite pattern[2] and discrepancies in the correlation of the high-angle reflections of *Y* and *tau* (Table I) seem to suggest that the accuracy of the diffraction data for both phases will have to be improved before one can say with certainty that *tau* and *Y* are identical.

Density considerations based on unit-cell volumes provide no clues for resolving the ambiguity of indexing and composition of the two phases, regardless of whether Ti_2S or TiN is taken as the correct formula. The same holds true for symmetry considerations. Systematic absences of the *hkl*-type were not observed in either set of indices, though the possibility of existence of systematic absences cannot be excluded for the (001) reflections, whose paucity does not allow definite conclusions to be drawn.

A detailed investigation of the two inclusion phases, preferably by single-crystal methods, would be of merit.

TABLE I.—HEXAGONAL AND TETRAGONAL INDEXING OF THE TAU AND Y PHASES.
(values of d in angstroms)

Tau						Y					
		tetragonal		hexagonal				tetragonal		hexagonal	
I*	d_o*	d_c	hkl	d_c	hkl	I†	d_o†	d_c‡	hkl	d_c†	hkl†
mw	2·77	2·775	110	2·777	100	w	2·788	2·773	110	2·777	100
vw	2·69	2·697	102	2·689	101						
vs	2·22	2·222	112	2·227	103	s	2·230	2·224	112	2·227	103
vw	1·963	1·961	200	1·971	104	vvw	1·977	1·961	200	1·971	104
w	1·859	1·858	004	1·865	006	w	1·870	1·858	004	1·871	006
vvw	1·736	1·734	202	1·742	105	vvw	1·740	1·735	202	1·743	105
ms	1·586	1·586	212	1·598	007	m	1·605	1·587	212	1·603	110
mw	1·542	1·544	114	1·541	112	w	1·550	1·545	114	1·548	106
m	1·381	1·386	220	1·378	201	vw	1·385	1·386	220	1·387	200; 107
mw	1·298	1·299	222	1·301	203	m‡	1·303	1·299	222	1·302	203
mw	1·212	1·210	223	1·216	116	wm	1·214	1·210	223	1·215	116
w	1·132	1·131	116; 215	1·135	108	w	1·135	1·135	215	1·134	109
vw	1·110	1·111	224	1·114	206						
vvw	1·052	1·047	206	1·050	210						
vw	1·045	1·044	322	1·045	211	vvw‡	1·047	1·049	206	1·045	211
ms	1·008	1·012	216	1·010	213	wm	1·0096	1·013	216	1·0103	213
vw	0·953	0·952	315	0·950	215	vw	0·9547	0·953	315	0·9550	1 0 11
						vw	0·9493	0·951	410	0·9502	215
mw	0·924	0·924	226; 330	0·926	300	w	0·9264	0·924	330	0·9262	209
						w‡‡	0·9249	0·925	226	0·9257	300
vw	0·916	0·917	331	0·915	216	vw‡	0·9142	0·917	331	0·9133	302
w	0·912	0·912	403	0·913	302						
RMS(d_o–d_c):		±0·0033¶		±0·0036				±0·0067		±0·0033	

* Taken from Ref. 1. † Taken from Ref. 2. ‡ May be overlapped by lines of another constituent (authors' remark, Ref. 2). ¶ First three values not considered.

as the pure compound, be it a sulphide or a nitride of titanium, may not be obtainable by synthetic methods.

Yours faithfully,

OSVALD KNOP.

Department of Chemical Engineering,
Nova Scotia Technical College.

Halifax, N.S., Canada.
7th December, 1957.

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AUTHORS' REPLY

The Editor, METALLURGIA.
Sir,

Comparison of the diffraction patterns of *tau*-phase and the *Y*-phase of Gemmill *et al*.[1] leaves little doubt that they are virtually identical. Ti_2S[2, 3] has been reported in the literature, but the patterns quoted bear no resemblance to that from *tau*-phase. We have found that the pattern can be indexed satisfactorily on the basis of several unit cells, including one similar to that quoted by Professor Knop. Owing to the difficulty in obtaining *tau*-phase free from other components and the possibility of changes of lattice dimensions due to small changes in composition, it is impossible to decide which unit cell is the correct one.

Furthermore, even when good agreement is obtained, there is no guarantee that the proposed unit cell is the correct one. For instance, several unit cells for *sigma*-phase were put forward, all of which were very different from the true unit cell.[4, 5]

As Professor Knop rightly states, single-crystal methods are needed, but the difficulty of obtaining a suitable crystal from extracted residues is considerable.

Yours faithfully,

J. F. BROWN, W. D. CLARK, and A. PARKER

Physical Chemistry Group, Imperial
Chemical Industries, Ltd.

Billingham.
23rd December, 1957.

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DOMESTIC METALLURGY

The Editor, METALLURGIA.
Sir,

Whether or not we were the "very large and self-satisfied group of ironfounders" referred to by your correspondent, Mr. Wood of the Midland Research Co., Ltd., we feel it necessary to put forward the manufacturer's viewpoint concerning the material used for bottom grates, etc., in domestic solid fuel appliances.

The material used for many years past has been a normal high-phosphoric grey cast iron, the same also being used to cast the remaining members of an appliance, vitreous enamelled or otherwise. Innumerable tests carried out in laboratories and under service conditions have shown that, provided the appliance is used as directed, i.e. correct burning rate, regular removal of ash, etc., the grate bars give an acceptable life. Unfortunately, many appliances are not used properly, the firebars in particular not being kept clear of ash, and this leads to an overheating of the cast iron, increased oxidation rates and rapid failure.

We ourselves have carried out many experiments on improving the performance of these parts of an appliance without increasing its cost, but have come to the conclusion that it cannot always be done. The use of an iron with $\frac{1}{4}$–$\frac{1}{2}$% chromium is quite definitely not the answer, since the high temperature oxidation rate is but slightly decreased with such a low alloy addition, and it is necessary to resort to the high chromium (30%) white iron before one gets any material improvement.

The use of such a material necessitates electric melting, segregation of scrap and a different foundry technique from that used with grey iron, with of course its attendant higher cost.

Recognising the economics of the situation and the difficulty of educating the user in the correct use of the appliance, most manufacturers will either fit a chrome iron grate and include it in the total cost of the appliance or will offer it as an alternative to cast iron at the cost of an extra few shillings.

And as a tribute to modern metallurgical knowledge, it is only a few shillings.

Yours faithfully,

C. C. HANSON

Birmingham, 6.
31st January, 1958.

Chief Metallurgist,
Radiation, Ltd.

Electric Melting Exhibition

NEW ideas in electric melting are to be shown to industry at an exhibition to be held by Birlec, Ltd., in conjunction with the London Electricity Board, at the Board's premises in Nelson Street, East Ham, from March 10th–21st. Principal exhibits will be the new Birlec-Morgan Electric Diecasting Furnace; the latest type of Birlec Tama low-frequency channel-type tilting furnace; and the Birlec Ajaxomatic holding and pouring unit for aluminium diecasting, operating under production conditions with a Herbert-Reed-Prentice $1\frac{1}{2}$ G diecaster. Also on show will be a scale model of the latest Birlec-Detroit rocking arc melting furnace.

Admission to the exhibition is by invitation, which can readily be obtained from Birlec, Ltd., or the London Electricity Board.

Uranium Find in Rhodesia

INDICATIONS of uranium have been discovered in the Wankie area of Southern Rhodesia, says Barclays Bank D.C.O. Anglo-American Rhodesian Mineral Exploration, Ltd., is reported to be negotiating with the owners of some of the claims pegged. Considerable pegging has taken place in the area, but in the rest of the Colony the search for uranium has so far proved unsuccessful. The Bank also says that the U.K. Atomic Energy Authority's plans to establish a uranium processing plant have been shelved.

Correction

ALUMINIUM AND ITS ALLOYS IN 1957

Readers are asked to note the following correction in the above article which appeared in our February issue:—

p. 80, the penultimate line in the first column should read :—

"on the American alloy 355 (LM16) with low iron content,"

Vacuum Melted Metals

Steel, Titanium and Zirconium in Production

ESTABLISHED before the French Revolution, Wm. Jessop and Sons, Ltd., is one of the oldest large steel companies in the United Kingdom. There is not, however, any evidence in the company of that trait of human old age, a tendency to live in the past. On the contrary, Jessops have in recent years shown particular interest in materials of the future, and have concentrated on the manufacture of steels and other special metals to particular requirements. With such a programme, extensive research facilities are needed, and almost two hundred physicists, metallurgists, chemists, engineers and other technologists are employed in the company's research department. Proportionate to its size, Jessops claims to have the biggest research department in the steel industry, its expenditure being many times the average for the industry as a whole.

Development of heat resisting steels began in 1936 and led to improved materials for steam turbines and for aero engine exhaust valves. Later work resulted in G.18B steel, which for about eight years was extensively used for jet engine turbine discs. Continued research produced steels better able to meet the advances in operating conditions, but G.18B is still suitable for certain stages, and each Britannia engine incorporates two G.18B discs and two H.46 discs. It is also of interest in the steam turbine field, as Jessops were asked to make a 5-ton rotor forging in G.18B for service in the highest-operating-temperature steam power station in the United States. Three research programmes of special importance at present concern vacuum melting and the production and fabrication of titanium and zirconium. Work in these fields is now bearing fruit, as they have all reached the works production stage.

Research and Development

The present Jessop research department had its beginnings in the early 1930's, when the existing works control analytical laboratory extended its activities to include some investigational work. In the early days development was slow, but there was a rapid increase in the size of the department when the company entered the field of high temperature steels and alloys. In 1950, Whiston Grange, near Rotherham, was adapted as a research laboratory for long-term investigation and development work, and some fifty members of the research department are accommodated there.

With the development of heat resisting materials, there has been a continually increasing demand for creep testing facilities, and Jessops now have in constant use over two hundred creep units—some at Whiston Grange and others at the works at Brightside—what is believed to be the largest installation in any industrial establishment in this country.

The Jessop research staff represents approximately 6% of the total employees of the company. This is a rather high figure compared with most other steel manufacturers, but with the wide range of Jessop products in steels, high temperature alloys, and special metals such as titanium and zirconium, research, development and control must play a large part in the company's activities.

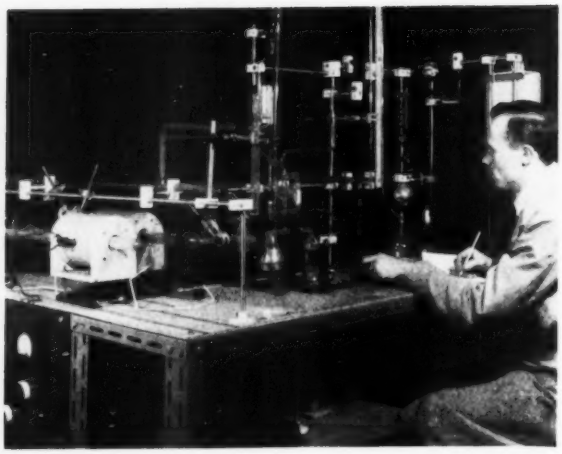
A view of part of the Jessop creep testing laboratories.

Vacuum Melting

Titanium must be melted in the absence of air, as the liquid metal is so reactive that it quickly forms a solidifying mass of titanium oxide. Even with quite small quantities of air present, enough oxygen may be dissolved to make the resulting metal hard and brittle. Similar considerations apply in the case of zirconium, which has a great affinity for oxygen and nitrogen, and these two metals are, therefore, vacuum melted. Other metals, including steels, can benefit from vacuum melting, as will be seen later in the section dealing with the production and properties of vacuum melted materials.

Pilot plant for vacuum arc melting is available at Whiston Grange, on which development work on both the process and the product has been carried out. An arc is struck between an upper consumable electrode and a water cooled copper crucible. In the case of steel, the upper electrode is a bar of material which was originally air melted, whilst titanium electrodes are made by welding together compressed titanium sponge blocks. For titanium melting, the current is in the region of 3,000 amp., and the arc voltage about 30. Ingots of 30–40 lb. weight can be produced in this plant. A new pilot plant is nearing completion, in which it will be possible to see what is going on, instead of having to rely on instruments, and in which it will be possible to add material such as scrap.

Vacuum melting can also be carried out in a 20 lb. high frequency induction furnace in which metal can be melted, alloying additions made, and the result cast into a mould—all within the vacuum chamber. This unit



Apparatus for determination of hydrogen content.

has been used to provide vacuum melted cast sticks as raw material for makers of precision castings by the lost-wax and similar processes.

The foregoing is not, of course, a comprehensive description of the facilities at Whiston Grange, but merely deals with those sections concerned directly with vacuum melting. To these should, perhaps, be added the residual gas analysis section, which has an important part to play in both the research and control aspects of these developments.

Production and Properties of Vacuum Melted Materials

Steels

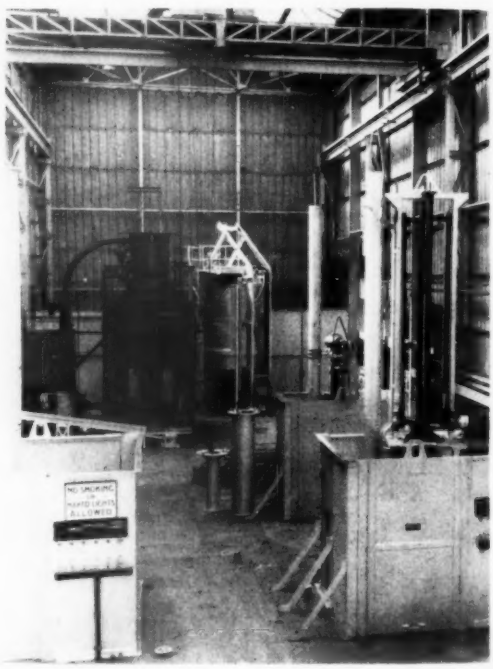
The main advantage of vacuum melting is essential freedom from non-metallic inclusions and impurities. This feature alone greatly reduces the danger of failure of highly stressed components, and gives improved ductility, notably in directions at right angles to the grain flow. Vacuum melting also allows closer control of the gas content and chemical composition of steels, reduces the melting losses of some elements, and improves the mechanical properties of certain existing alloys. Furthermore, the ability to produce alloy compositions which cannot be melted by conventional methods opens up new fields for the development of alloys with properties superior to those now available for specific applications.

A number of Jessop Vacumelt steel qualities have been evaluated on the basis of mechanical properties in comparison with the air melted properties. The tests have included high speed steels, bearing steels, and stainless, ferritic creep resisting, and austenitic steels. Tensile, creep and fatigue tests have been carried out, and vacuum melted steels have given results at least as good as, and in many cases greatly superior to, those for the corresponding air melted steels.

Taking two specific examples, a creep resisting steel disc and an 18/4/1 high speed steel, the following results were recorded.

Jessop H.53 is a 12% chromium creep resisting alloy. In the air melted condition, and when hardened to a tensile strength of 70–75 tons/sq. in., the longitudinal ductility is of the order of 15%, while the transverse ductility in the centre of a large forging may be as low as 5%. Tests from a 24 in. diameter Vacumelt H.53 disc forging at the same U.T.S. level showed longitudinal elongations of the order of 20%, with central transverse elongations of 14–19%, i.e. three times the ductility in the worst position. A full and careful microscopical examination of this forging was made, and the largest non-metallic inclusion to be found was only 0·003 in. in length.

Jessop J.3 is an 18/4/1 type high speed steel, which is being used among other things for high temperature bearings. In the air melted condition, this alloy is comparatively free from impurities, and has a room temperature fatigue strength of ±40 tons/sq. in. for 5m. reversals. It is well-known that an extremely high grade of purity is required in ball races to ensure long life, and vacuum melting this alloy produced a super grade of cleanliness. Vacuum melting also increased the fatigue strength from ±40 to ± 43 tons/sq. in.



A general view of the vacuum arc melting plant.

From the foregoing it will be seen that vacuum melting offers distinct advantages for steels for certain purposes. Such applications are too numerous to list, but reference may be made to a few of them. One obvious example is that of ball bearings and ball races, where the life of such components may be increased fourfold. In the aircraft industry too, where high temperatures and high stresses are competing with the ever increasing necessity of prolonging periods between maintenance, vacuum melting would offer advantages. Equally highly stressed components in steam generating plant, fine gauge quality steel wire, and components requiring a perfect polished finish—such as plastic moulds, extrusion dies, etc.—are also examples of applications which can suitably employ vacuum melted steel in their manufacture.

The methods adopted by Jessop for vacuum melting

are: (1) the consumable electrode arc melting process: and (2) the high frequency induction melting process. In the former, air melted material is used as an electrode and remelted under vacuum in similar, but larger, plant to that at Whiston Grange. At present, ingots weighing 20 cwt. can be produced by this method, but when the new plant is installed the capacity will be doubled. The high frequency induction melting process is at present confined to the 20 lb. capacity unit at Whiston Grange, but a 600 lb. plant has been ordered for installation at the Brightside works. One advantage not mentioned earlier which the vacuum arc melting process has over both air melting and vacuum induction melting is that the resulting ingot is relatively free from pipe. On the other hand, the arc process is unsuitable for alloys in which the production of an air melted electrode is impossible.

Titanium

As has been mentioned earlier, melting in the absence of air is essential for titanium and its alloys, because of the reactive nature of the metal. On the other hand, its corrosion resisting properties at normal temperatures are good, and titanium is finding application in marine and chemical engineering for that reason.

The mechanical applications of titanium arise from the combination of strength and lightness to be found in its alloys. Although its density (0·163 lb./cu. in.) is higher than that of aluminium (0·096 lb./cu. in.), tensile strengths obtainable range from about 27 tons/sq. in. in the commercially pure form to 80 tons/sq. in. when alloyed and heat treated. Such values in a material which has a Young's modulus in the region of 7,500 tons/sq. in. at room temperature make it attractive for certain applications, particularly in aircraft engineering. It is also advantageous for certain parts of mechanical devices which are oscillating rapidly, and where it is desired to reduce the inertia. Certain of the alloys have been developed for elevated temperature operations.

Jessops are now marketing a number of alloys of titanium under the trade name of Hylite. Two of these, Hylite 10 and Hylite 15 are commercially pure grades of titanium with different hardness ranges, Hylite 15 having the greater tensile strength. Hylite 10 has a maximum hydrogen content of 0·013% and is suitable for applications where maximum formability and weldability are required, and for applications involving corrosion resistance. The tensile strength is around 27 tons/sq. in.

20 lb. vacuum induction melting furnace.

Hylite 20 is a 5%-aluminium, 2½%-tin alloy with medium tensile strength (55 tons/sq. in.), good weldability, and non-embrittlement under service conditions. It has a maximum hydrogen content of 0·013%, and is recommended where a higher tensile strength is required than can be provided by Hylite 10 or 15.

The next grade, Hylite 30, is a 2%-manganese, 2%-aluminium alloy of medium strength (45 tons/sq. in.), which again has a maximum hydrogen content of 0·013%. This material is recommended for forgings, etc., where reasonable strength is required.

Hylite 40, with a higher tensile strength (65 tons/sq. in.) and greater creep resistance than Hylite 30 is a 4%-aluminium, 4%-manganese alloy with a maximum hydrogen content of 0·013% and excellent properties as forgings or bar material. It is suitable for many applications, such as a compressor disc or blade material.

Slightly superior to Hylite 40 in tensile strength, Hylite 45 is a 6%-aluminium, 4%-vanadium alloy with 0·013% maximum hydrogen content. It is recommended for forgings and is particularly suitable as a high strength sheet material, as it can be formed and welded in its most ductile condition and subsequently heat treated to obtain optimum properties.

The last alloy in the series is Hylite 50, a complex titanium-base alloy containing molybdenum, silicon, tin and aluminium. This material has a tensile strength of 80 tons/sq. in. but it has been specially developed to have good creep properties, for the highest duty service as a compressor disc or blade material. It is supplied with a maximum hydrogen content of 0·015%, and prolonged exposure in hydrogen at temperatures up to 120° C. has not resulted in pick-up. Even at 300° C., after 1,000 hours duration the hydrogen pick-up was no more than 20–30 parts per million.

The large scale production of titanium and its alloys is carried out in a furnace which is similar in principle to that used for development work at Whiston Grange. The titanium powder or sponge is mixed uniformly with the alloying metals and pressed in a 2,000 ton hydraulic

Ultrasonic inspection of large titanium slab.

press into a half octagonal prism, 18 in. long × 9 in. across flats. These pressed blocks are welded together to form an electrode, being staggered longitudinally so that no joint goes completely across the electrode. Argon arc consumable electrode welding is used, and the construction is sufficiently strong for its purpose. The welded structure is made the consumable electrode in the arc melting furnace and in this way is converted into an ingot, which may have a diameter of 16 in. A double melting technique is adopted for titanium, the ingot from the first melting acting as the electrode for the second melting to produce an ingot with a diameter of up to 20 in. This procedure ensures a completely homogeneous alloy with the minimum impurities present, the total hydrogen content of a typical melt being of the order of 160–170 p.p.m.

The plant was designed and built by Jessop engineers in 1954, as a private venture at a cost of £300,000. Ingots weighing 1 ton can be produced at present, but this will be doubled when the new plant comes into operation. With the cut-back in the Defence Programme, additional outlets for titanium and its alloys have to be found, and the company are actively pursuing markets both in the United Kingdom and overseas in such widely diverse fields as aeronautics, armaments, marine engineering, chemical engineering and medicine. In recent years the price of titanium has fallen appreciably, but the defence cuts have, for the time being at any rate, halted that fall. In any case, titanium is unlikely to become a cheap metal, with production methods envisaged at present, as it is unlikely that a sponge price below 10s./lb. will be achieved.

Zirconium

Zirconium production is akin to that of titanium, in that the metal has a great affinity for oxygen and nitrogen, and the melting process must therefore be carried out under vacuum. It is not a light metal, having a density a little lower than steel, and its main application is in the atomic energy field. Zirconium and its alloys have good corrosion resistance and a low neutron absorption value, and hence can be used to advantage within the reactor. Alloy research is going on at present in collaboration with the Atomic Energy Authority, and it is expected that melting of larger quantities will be undertaken when industrial groups requiring the metal decide what alloys and shapes are most suitable.

Changes at the N.P.L.

DURING 1957 a comprehensive review of the organization and programmes of work of the National Physical Laboratory was carried out by a committee of the Research Council of the Department of Scientific and Industrial Research. As a result, the Research Council have now approved certain changes in organization, which will become effective about the end of March.

One of the most important is a reorganization of the work at present carried out in the Divisions of Electricity, Metrology and Physics and in the Test House. These four units will be replaced by three new Divisions to be called Standards, Applied Physics and Basic Physics.

The Standards Division will be responsible for all fundamental work on standards of length, mass and time; of electrical and magnetic quantities; and also of temperature. Basically, it will consist of the present Metrology Divisions, expanded to include certain work on standards now carried out in the Electricity and Physics Division. The Superintendent will be Dr. H. Barrell, at present in charge of the Metrology Division.

The Applied Physics Division will be responsible in general for work in the field of classical physics of fairly immediate value to industry (but excluding optics, all of which will continue to be done in the Light Division), The principal areas covered will be electrotechnics, acoustics, heat and radiology. Test House, which hitherto has come under Administration, will become an integral part of this Division, but will preserve its indentity under Mr. H. Bowley. The Superintendent of the Applied Physics Division will be Dr. B. Wheeler Robinson, who is in charge of the present Physics Division.

The Basic Physics Division will be responsible for pioneering developments in certain branches of non-nuclear physics which have potential industrial applications in the less immediate future. The post of Superintendent of this Division has recently been advertised.

The Council recently recommended the creation of a new post of Deputy Director, and Dr. E. Lee (at present Director of Operational Research in the Royal Naval Scientific Service) has been appointed to it. He will take up his duties on 31st March. The Deputy Director will take the main responsibility for work in the Laboratory which is closely geared to immediate demands from industry.

Conference on Welded Steel Structures

An All-Day Conference, organised for Friday, May 16th, 1958, by The West of Scotland Iron and Steel Institute, has as its subject: "Metallurgical and Engineering Aspects of Weight Saving in Steel Structures." The following papers have been promised:

Introductory paper by PROF. A. S. T. THOMSON, Glasgow.

"Plastic Theory of Design," by DR. M. R. HORNE and MR. T. M. CHARLTON, Cambridge.

"The Application of Cold-Formed Sections," by DR. R. KENEDI, and MR. W. SHEARER SMITH, Glasgow.

"The Application of Low-Alloy High-Tensile Steels," by MR. I. M. MACKENZIE, Motherwell.

"The Application of Tubes to Structures," by MR. G. B. GODFREY, London.

"Bridges and other Structural Work with Special Reference to Welded High-Tensile Steel," by MR. G. ROBERTS, London.

"Weight-Saving in Military Steel Bridges," by MR. A. E. LONG.

A paper on Light Gauge Sections and Roll Design by MR. W. G. KIRKLAND, New York.

There will be a registration fee for the Conference and it will also be possible for persons not attending the Conference to purchase a set of preprints of the papers. The final details are not yet available, but a circular is being prepared and may be obtained from the Secretary of the Institute, 39 Elmbank Crescent, Glasgow, C.2.

New Telephone Number

AN additional exchange line has been connected at the Aberdeen Branch Office of British Insulated Callender's Cables, Ltd. The new telephone numbers are: Aberdeen 53351/2.

The Johnston Buffer

An Item of British Steel Foundry History

ON June 25th, 1895, George Johnston, then living at 65, Eastbourne Street, Liverpool, but ordinarily of Durban, Natal, applied for a British patent for an improvement in automatic central buffers and couplings for railway use. Johnston took the ordinary central buffer as used in America and the Colonies and introduced into it a casting (*B*) (Fig. 1), provided with a stirrup (*C*), and spring catch (*D*). When the link was pressed in, it was guided by the stirrup and the catch was drawn back, allowing the coupling pin (*E*) to fall down through the link and through holes in the casting and the buffer box above and below. The coupling pin passed through a casting applied to the outside of the buffer box.

This pin had a projecting arm over which a slide link (*H*) passed to prevent the pin from being entirely drawn out and to keep it from turning. A pin passing through both casting and buffer box and also through the subsidiary casting on top of the buffer box held the entire assembly together. To raise the coupling pin when required, a chain from an eye bolt on a horizontal cross bar, worked from each side of the carriage, was used. The bar was provided with feathers working on its bearings or guides, and was so arranged that by turning the bar the feathers did not lock, and the bar could be pulled out of either side.

By turning the bar with the crank handles downwards, however, the feather locked in whatever position it occupied. Thus, by pulling or pushing the bar in either direction and turning it with handles down, the pin was withdrawn from the link, leaving the link free.

The link acted by entering the bull mouth of the central buffer, and was guided by the sloping sides and the stirrup into exact position for the pin to fall. The link pushed back the spring catch on the carriages coming together, so leaving the pin free to fall through the link and thus fasten the buffer. The moment, however, that the horizontal bar caused the pin to be withdrawn from the link, so separating the two carriages, the spring catch flew into position, and prevented the pin from again falling until this or another link was again inserted, when the weight of the pin on the pushing back of the catch by the link caused the said pin to fall and locked the coupling.

Before the complete specification had been deposited, Johnston wrote to Edgar Allen & Co., Ltd., Sheffield steel manufacturers and founders, and later called to see them (October 14th, 1895). He had already in his pocket, he claimed, an order for 200 buffers of his new design for the Natal Railways, and had sent this to Webster, Steel & Co. of London, who, it seems, had returned it to Durban, probably not knowing, as merchants, whom they could entrust with its execution.

Johnston suggested that the firm (Edgar Allen & Co., Ltd.) should take a fourth share of his patents, which, he considered, would induce them to " push " his buffer more actively than if they were merely sole licencees. On December 3rd, he had an interview with the Directors of the Company, and the following day the firm wrote to him : " We trust the agreement will soon be drawn up

A Central buffer
B Casting
C Stirrup
D Spring
E Coupling pin
F Holes for pin
G Subsidiary casting
H Slide link

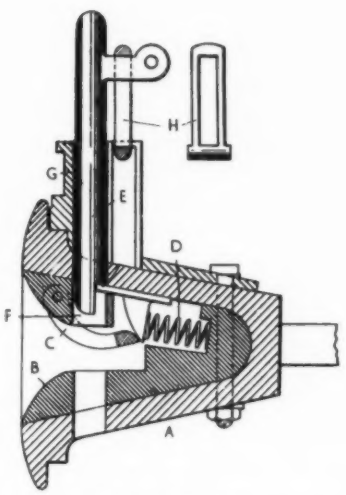


Fig. 1.—Johston's first patent buffer.

under which we are to have the sole right to make your patent buffers, especially as orders are coming forward for rolling stock to be built in this country and fitted with these buffers. We have been waiting the draft for several weeks. The question of purchasing a fourth share of your patent rights was not, of course, in your original proposal to us, and, as you intimated yesterday, it is not an essential condition of the agreement to manufacture the buffers."

Although the patent had still to be accepted, the firm had evidently begun to manufacture the buffers, because in a letter to their representative in South Africa, they wrote on January 17th, 1896, " We shall most probably send some off to Durban this month. Johnston was here yesterday and shewed us a letter he had had from Cammell's man, John Toder, who had been to Durban and seen Mr. John M. V. Brown and Black, the loco. foreman. Toder wanted to see Johnston about making his buffer . . . The 200 buffers are ordered by James S. Wylie, solicitor, 333 Smith St., Durban, with whom Brown and Johnston are associated. Johnston says a young Steel is partner with Wylie. No doubt Webster, Steel & Co. are sore about our getting this buffer into our hands . . . Johnston is taking out another patent for a modification of the 1st patent. We have made 2 to this new design and are sending them at once to Mr. Wylie."

The 200 buffers referred to in this letter are obviously those mentioned by Johnston in his first interview with the firm, and it appears that this order had evidently been transferred on Johnston's instructions to Edgar Allen's. The presence of " a young Steel " in Wylie's office doubtless explains the original despatch of the order to Webster, Steel & Co.

Johnston's modification of his first patent, referred to above, was not applied for until May 15th, 1896, and we

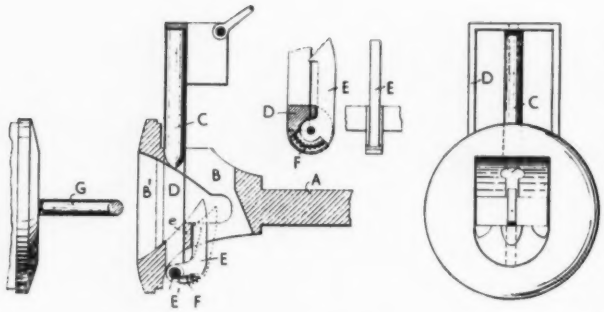


Fig. 2.—Johnston's third patent buffer.

shall deal with this in due course. Meantime, the two buffers of the new design were despatched to Wylie from the Imperial Steel Works on January 21st, 1896, together with six pieces about 7 in. long by 1 in. sq., cut from locomotive wheel centres and other castings to represent the quality of material from which the buffers had been made. "Tell the foreman smith at Durban to have them tested as he thinks best," the firm wrote, giving examples of appropriate tests that could be carried out. The letter adds: "We shall shortly be in a position to turn out 50 *sets per week*" (of the 200 original buffers ordered). "The cost and preparation of moulding boxes, machines, etc., will be great to effect this turnout, but we hope the early future, will, in the way of orders, justify this step."

By January 27th, the firm had still received no formal agreement, and on this date they submitted detailed suggestions, one of which was a royalty of £2 10s. 0d. per buffer for all buffers sold by them after payment had been received from the buyers.

On May 2nd, Johnston's original patent was formally accepted (No. 12,270). His new specification was designed to overcome a defect objected to in the first patent, namely that as the buffer head was rigidly united to the buffing or drawbar, the former remained at right angles or nearly so to the head stock of the vehicle. The buffers being thus rigid, the buffing action between the two vehicles on a curve was mainly confined to one side, or the other of the buffer faces. This was liable severely to strain the vehicle, and had other disadvantages.

Johnston now pivoted the buffer box to or coupled it to the buffing and drawbar so that the former was not rigidly united to the latter, but could turn on its axis to facilitate the alignment of the buffers of adjacent carriages when these were on curves. This arrangement enabled the buffer faces to be made flat, with the buffing action distributed over the whole face of the buffer. The end of the bar abutted against a solid abutment on the buffer head so that the entire pressure on the buffer was received by the end of the bar instead of on its pivot. An arrangement was provided for causing the buffer to resume a position parallel with the bar when the vehicle was detached or travelling in a straight line. This took the form of a spring button or stud on the end of the bar, having a spherical face engaging a depression in the buffer box. It was so arranged that when the spring button engaged the depression in the buffer box or head, the head was held in line with the bar.

By the exertion of a little force, the buffer head could be turned on one side or the other, so that it would at once resume its position when the lateral turning force was removed. Mechanical means of engagement with the jaw were provided.

Another item in this application was the use of a pivoted lever on which was a horizontal crossbar. This, when raised, was adapted to engage the projecting arm and raise the coupling out of engagement with the link. The lever dropped down after the force which had lifted it was removed, the coupling pin being left raised until two vehicles were again brought together.

The months had gone by monotonously, but still Johnston could not be brought to sign a definite agreement, and on September 11th, 1896, Edgar Allen & Co., Ltd., received a letter which caused them great consternation. This read: "after careful consideration, he" (Mr. Johnston) "has determined not to give a licence for you to manufacture and sell his patent buffer." This drew a vigorous protest, with the result that further negotiations began. Johnston was evidently contemplating a return to South Africa, and some of his stipulations were obviously unacceptable.

For example, he wanted royalties to be paid on buffers that had been rejected for any reason whatsoever. He wished to be consulted in all instances before prices were quoted, which in view of his plans to return to Durban was absurd. He wanted his agents to have the right to inspect the buffers, even during manufacture, to which the firm objected because they were not willing to have people wandering over their foundry. He also wanted guaranteed delivery of at least 120 buffers a month, which again was absurd, because in many instances the wagon makers would not accept delivery until they needed the castings. The firm pointed out that actually they were now in a position to supply 200 a month, but they disliked being tied to a specific figure. Another stumbling block was that the firm wanted Johnston to protect his patent from infringement, which he seemed reluctant to do.

By September 30th, 1896, these points had nearly all been settled in the firm's favour. On October 12th Johnston called at the works to ask what provision could be made to test the probability of the Netherlands Railway (Transvaal) objecting to take buffers of English make. (The troubles in South Africa were already beginning). There was undoubtedly a strong feeling in the Transvaal against using English manufactures, but the firm pointed out that if the Cape and Natal Railway Cos. adopted his buffer, it was still most important that all should be made at one place to ensure complete interchangeability of parts.

On October 16th, Johnston wrote complaining that the firm was "procrastinating," which drew the sharp rebuke "I am indeed surprised you should allow yourself to pen such words for you must know we are not the culprits! Further, you know there are two or three matters to be cleared up, and only waiting your attention."

Logic, reason, patience, argument, could not pin down the elusive inventor, and on October 29th he is again raising fresh difficulties. This caused the firm to write: "It appears to us that no sooner has Mr. Johnston made up his mind than he goes away and turns completely round. We are tired of this sort of thing and we are quite sure Mr. Wylie and Mr. Brown in Natal would not approve of such conduct. We must insist on the Licence for South Africa and British Possessions being completed

forthwith on the terms already definitely agreed to by Mr. Johnston. We quite agree that 'Mr. Johnston honestly wishes' us to execute all Buffer orders, but he has a remarkable talent for looking at his own side of the subject and not at ours."

South African methods of business in those days were not all that might be desired, and the firm wrote to Johnston on November 3rd, 1896, to this effect: "It would appear that there is a sort of conspiracy going on against your buffers and against ourselves in this matter, but do not take any action in this country at present because we wish to have an interview with the Agent General on this subject and it will not do for you to be mixed up in the matter at the present stage."

On November 10th, Edgar Allen's wrote to their solicitor, George J. Simpson, of Sheffield: "if we take out a licence for Gt. Britain and all British Possessions, we could stop Transvaal rolling stock going into Natal and Cape Colony if fitted with Johnston's buffers not made by us. Is this the law? If so there will be no harm in our agreeing to the Transvaal being left out."

Trouble still continued, and on November 11th, the firm wrote. "Mr. Johnston may be annoyed at our reference to his mode of action, but we have, in our opinion, every justification for using much stronger language than we have hitherto used in connection with these negotiations. From the beginning, Mr. Johnston has led us to understand that we were to have the sole right to make his buffers, and then, at the last moment, he says he must have 'a free hand for the Transvaal.' Notwithstanding that, we repeat that Mr. Johnston, at the consultation we had with him here . . . agreed to include the Transvaal in the licence on the understanding that if the Transvaal Government would not give us the orders *direct*, they were to be negotiated through some foreign firm who would be acceptable to them. Now Mr. Johnston turns round and says he will not include the Transvaal in the licence. We should like to know what is the language which would properly describe this conduct, or what language Mr. Johnston would use if he were in our position?"

On November 21st, 1896, Johnston's second patent (No. 10,501) was officially accepted. He had already filed an application for a third patent on July 8th, 1896. Acrimonious correspondence with him still went on, and by November 30th, his colleagues in South Africa had been drawn in. The firm wrote: "On account of the way in which Mr. Johnston has delayed the completion of the licence and the consequent uncertainty of our position in the matter, we have not, of course, taken any steps to bring the buffers before our friends, nor have we got out drawings, circulars, and models, etc. For this valuable time lost Mr. Johnston has only himself to blame." Evidently the South African influence was exercised in the firm's favour, and the licence was drawn up and formally signed in February, 1897. Johnston still talked of agreeing to include the Transvaal.

Johnston's third patent application covered a new type of coupling link (Fig. 2). Slots or openings (*B*, *B'*) were formed in a central buffer (*A*). The coupling pin (*C*) had a projecting arm to enter the slot, as well as a saddle (*D*) that, keeping the pin from turning, enabled it to slide freely up and down. It prevented the pin from being drawn entirely out, however. A catch (*E*) was provided at the bottom of the saddle and mounted on a transverse spindle (*E'*). This was adapted to engage an abutment (*e*) on the buffer head, the catch being spring-controlled (*F*). Consequently, when the draw link (*G*) was pressed into the buffer head, the draw link, pushing back the catch, disengaged it from the abutment, allowing the coupling pin to fall down through the link and through the holes in the buffer head above and below. The coupling pin was raised when required by a chain coupled to the shackle. A stop prevented the catch from turning too far forward.

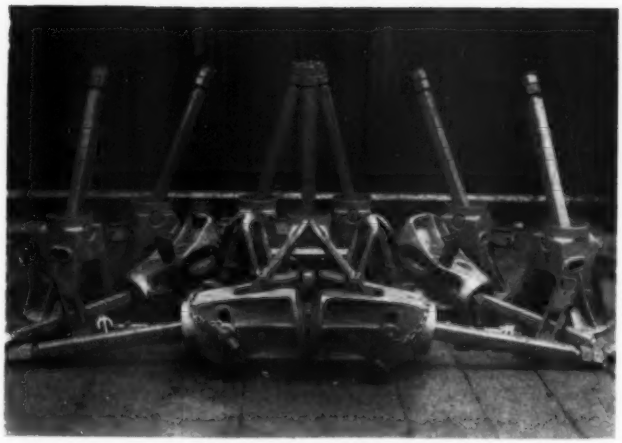


Fig. 3.—A group of Johnston buffers.

The coupling link acted as follows. When two vehicles came together, the draw link automatically entered the bell mouth of the central buffer and was guided by the chamfered mouth thereof into the exact position for the coupling pin to fall. The link, pushing back the catch from engagement with the abutment on the carriages coming together, left the pin free to fall through the link and thus fasten the buffers. The moment, however, the pin was raised from the link and the two carriages separated, the spring catch flew into position, engaging the abutment on the buffer, and prevented the pin from again falling into position until this or another link was again inserted, when the weight of the pin and its saddle on the pushing back of the catch by the link caused the pin to fall and lock the coupling link. The weight of the pin together with its saddle, pressing through the arm on the link, held the link in a horizontal line, so that when two buffers were brought together, the coupling link of one buffer would approach the mouth of the other buffer horizontally, and furthermore, the mouth of the buffer being chamfered ensured that the link would enter it even if one buffer were in a lower plane than the adjacent buffer.

Johnston had evidently patented his buffer in the United States, because on January 17th, 1898, Edgar Allen & Co., Ltd., sold a number of his buffers to an American firm for rolling stock they were supplying to the Orange Free State. The U.S. Patent was not being worked in that country at this date, and there was some doubt at the time whether the importation of these buffers into the United States in any way affected the validity of Johnston's patent there. Johnston's third patent (No. 15,151) had been officially accepted on June 5th, 1897, and between this date and July 19th, 1898, he evidently returned to Durban.

Facts and figures regarding the supply of Johnston buffers are difficult to come by after this lapse of time, but it is clear from a royalty statement still existing that for the quarter ending June 30th, 1898, royalty was paid on 100 buffers, supplied to the Jackson and Sharp Co. of London, and Alexander Shanks & Sons Ltd. of Arbroath (two only). The buffers were sold at £6 each, and the royalty paid was £2 each on 98 buffers, and £2 10s. 0d. each on the two for Arbroath. From a statement dated July 22nd, 1898, it appears that the buffers were also patented in India and Ceylon. Buffers for 100 wagons were supplied to James Brown of Durban in 1897, and 200 buffers in the same year to the Birmingham Wagon Co.

Johnston appears again in 1900, when on May 24th, he submitted yet another application for a patent, the object of which was mainly to provide an apparatus that would lift the coupling pin of the central buffer and hold it in the raised position when required, so as to allow the draw link automatically to enter the bell mouth of the buffer when two vehicles come together, and prevent, during shunting operations, the automatic falling of the coupling pin to fasten the buffers. This patent was formally granted (No. 9572) on December 8th, 1900, by which time Johnston was back in Natal.

The mechanism for automatic coupling of the buffers did not prove successful and was dropped in favour of later patents. The first of these was accepted on July 20th, 1905 (No. 20,464). In the older type, the drop pin had holes forming the top and bottom bearings so far apart that a pin of moderate diameter was liable to bend when the tension on the coupling links became excessive. Patent No. 20,464 was designed to obviate this tendency and shorten the pin bearings by providing projections, lips or brackets inside the bell mouth of the buffer head.

On April 27th, 1907, a further patent (No. 9970) was accepted designed to arrange the buffer in such a way that it was not rigidly united to the buffing and drawbar, but was pivoted so as to be capable of moving in a horizontal arc upon the pivot pins. This meant that the buffers of adjacent vehicles would always be in line when coupled up with the faces in touching contact, no matter whether the vehicles were on a curve or not, and as the buffers were made flat, the buffering action was distributed over the whole face of the buffer. The central buffer had a bell mouth and a coupling hook projected from the front face of the buffer.

When this improvement was patented, Johnston was back at 16, Balmoral Road, Fairfield, Liverpool. In the following year, his last patent, No. 8362, was accepted (April 9th, 1908). This was merely a method of improving and strengthing the construction of the coupling for traction purposes. For example, one aspect of the invention provided at the ends of the two arms of the body a buffer face grooved at its rear to clasp the ribs of the body, a draw hook at the other side of the centre line, and the arms spaced at such a distance apart that both draw hooks were completely contained in the space between the arms when two vehicles were coupled together.

The only genuinely successful Johnston buffer was that in which the crosshead slid in a guide box, the buffer shank abutting against the solid part of the inside of the crosshead. The buffer head and shank of this type were kept central by means of a vee piece and a rectangular spring. The South African Railways continually complained of these rectangular springs, which broke because the design caused them to be compressed on one side only, according to which way the head moved.

In consequence, on February 25th, 1915, Patent No. 16,492, was granted in the names of Cecil Watson of Roslin, Grove Park, Lee, London, and Hugh Donald Boyd of 40, Glencoe Road, Sheffield. This covered an improved vee piece designed by H. D. Boyd, then a member of the foundry staff of Edgar Allen & Co. Ltd., and now one of its directors. This invention enabled the spring to be evenly compressed. The patent was first applied for on July 10th, 1914, when the original Johnston patent was on the point of expiring. About 200 buffers were made with this improvement, the expectation being that by this means the firm would retain monopoly of supply. The South African Railways were, however, determined to break the monopoly and manufacture these buffers in their own workshops.

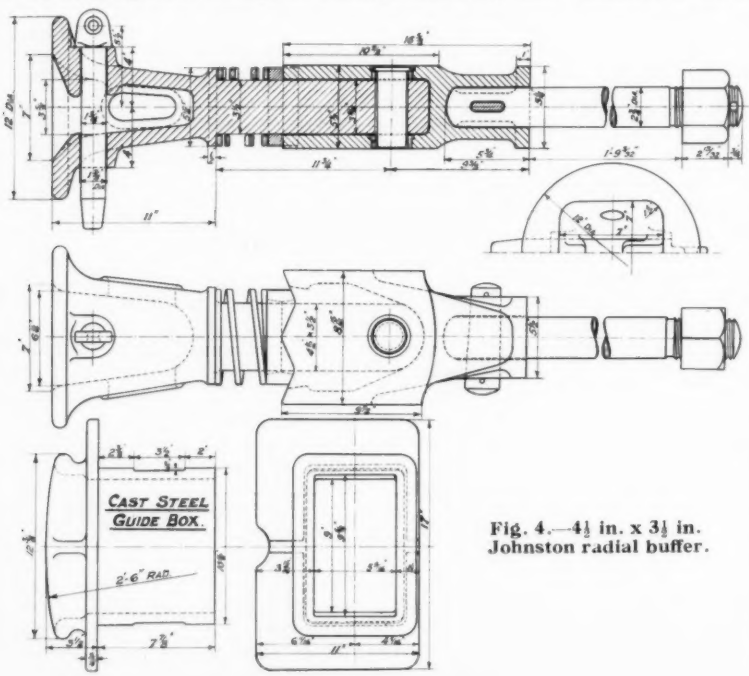


Fig. 4.—4½ in. x 3½ in. Johnston radial buffer.

The two drawings, Figs. 4 and 5, show the type of buffers actually manufactured by the firm. Fig. 5 also show Hyde's patent abutment, a simple contrivance consisting of a piece of steel cast inside the buffer head to support the coupling pin, thus preventing the pins from bending. Hyde was a blacksmith in South Africa and received 2s. 6d. royalty on every buffer made by Edgar Allen's with this abutment. Later (about 1926) he was employed by Messrs. Vickers Armstrong, Ltd., at their Barrow-in-Furness works.

By modern standards the Johnston buffer was somewhat crude. It was not what was known as a tight coupling. In other words, it resembled the method used for wagons in this country at the present time. There was always about an inch of slack to take up between each carriage when starting.

The buffer also failed to find favour outside South Africa and Rhodesia. The shanks, it should be noted, were forged under the hammer, the type with a crosshead having a rolled steel shank "cottered" into it. For a time the South Africans made the buffers as forgings rather than as castings. Today they use the Atlas and Alliance types of automatic couplers, and even Rhodesia, to which Edgar Allen's still supply a few Johnston buffers, are gradually adopting the more modern types as well.

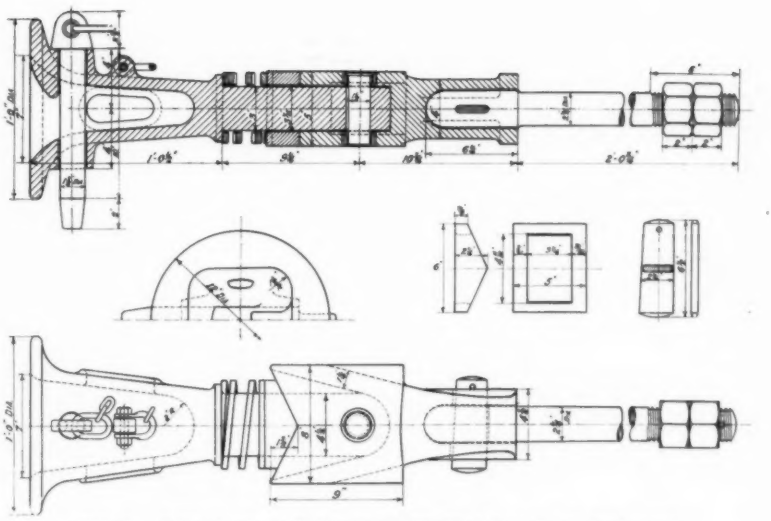


Fig. 5.—Johnston's radial buffer with Hyde's patent abutment.

Up to the beginning of World War I, Edgar Allen & Co., Ltd., regularly made and shipped to South Africa and Rhodesia many thousands of Johnston buffers. When the patents expired and foreign competition, combined with home manufacture in South Africa, stopped the flow of orders, the output of this type of buffer at the Sheffield works declined to modest dimensions, and virtually ceased in 1916.

A New Theory on Stress-Corrosion Cracking

A NEW theory on the phenomenon of stress-cracking in metal structures is based on the discovery that minute crystals resembling delicate plates tend to grow from the surface of stainless steels when the metal is stressed and exposed to corroding atmospheres. The newly discovered crystals are described as "submicroscopic platelets of chromium oxide" and they form on strongly stressed stainless specimens which are exposed to atmospheres containing traces of negatively charged chlorine atoms.

The discovery, made by Dr. E. A. Gulbransen, advisory chemist at Westinghouse Research Laboratories in Pittsburgh, is likely to lead to a better understanding of the fundamental mechanisms involved in corrosion. Although it is known that failure of metal structures is often due to a combination of chemical corrosion and internal or applied stress, and that the failure can often be initiated by a relatively mild corrosive substance, little information is available which explains how the phenomenon, known as stress-corrosion cracking, operates.

A possible explanation based on the new discovery, is that the growth of the platelets on the surface of the steel may lead to a "chemical cutting of the metal." Minute crevices may grow downward into the metal surface as the platelets thrust themselves above it. This may lead to a concentration of stress at the base of the crevices and eventually to failure.

One of the experiments leading to the new theory involved subjecting a small piece of stainless steel wire 0·009 in. in diameter to a carefully controlled atmosphere of oxygen and water vapour at a temperature of 1,100° F. (600° C.). A small disc of stainless steel, 0·005 in. thick and having a hole 0·006 in. in diameter, was sometimes substituted.

After corrosion by the hot atmosphere, the wire or disc was examined and photographed on an electron mieroscope. A typical sample was found to erupt with "billions of oxide whiskers." Only about one or two millionths of an inch in thickness, these whiskers grow to a height 300–400 times their diameter. Their density is about six billion per square inch of metal surface.

Completely unexpected changes occur, however, when a sample is pre-stressed and a trace—less than five parts per million—of chloride ions is added to the atmosphere. Instead of long, thin filaments, rows of thin, upright, parallel plates grow along a definite crystallographic direction. Their thickness is placed at 0·00005 in., or less. Analysis shows them to have the characteristic structure of an oxide of chromium, normally present in stainless steels; in fact each tiny plate appears to be a single crystal of chromium oxide (Cr_2O_3).

Although it has been known for some time that the chloride ion is a major factor in the stress-corrosion cracking of stainless steels, the mechanism has remained unexplained. The knowledge that chloride ions stimulate growth of crystals in strongly stressed metal, and that these crystals tend to crack the metal and lead to ultimate failure, may eventually enable researchers to prevent such failures.

From *Materials in Design Engineering*, **47**, (1), 150.

Ultrasonic Cleaning

IF ultrasonic energy is injected into a liquid, it gives rise to cavitation on the surface of any object immersed in it. The rapid generation and collapse of a multitude of small bubbles has the effect of rapidly removing any loose matter from the immersed object, without damaging its surface in any way.

This new cleaning technique has many important applications in the field of metal treatment and finishing, as well as in the production and processing of metal parts. The strength of soldered, brazed and welded joints, the adhesion of plating, lacquer, enamel and similar finishes and the operation of many precision components depend to a large extent on the thoroughness with which preparatory cleaning can be carried out. For such work ultrasonics show a distinct advantage, while the cleaning of blind holes, cavities and intricate shapes can often be done in no other way. The elimination of scratches due to scrubbing also renders ultrasonics ideal for cleaning dies, moulds and other metalworking tools. By mechanising the feed of parts into and out of the bath, ultrasonic equipment is easily incorporated into production lines, where its speed, uniform action and compactness can confer important benefits.

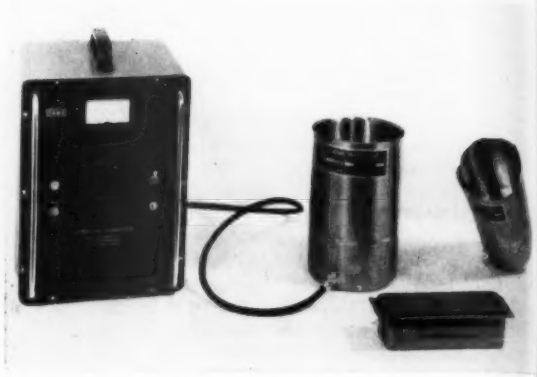
Standard Equipment

In the past, a frequent obstacle to the wider use of ultrasonic cleaning has been the lack of standard equipment, since the initial cost of units tailored for a specific purpose has naturally been high. As may be seen from the illustration, the standard Dawe Type 413 ultrasonic generator now provides a convenient means of driving a series of standard transducers. Drawing only about 250 W. from the mains, each generator provides power at 38 kc. s., sufficient for transducers having a total area of 30–40 sq. in. This area is ample for one standard 1-gal. tank or for two ½-gal. tanks, each tank having the transducer built into the base. Alternatively, standard fully immersible transducers ($2\frac{5}{8}$ in. × 8 in. × 3 in. high) are available to convert existing cleaning tanks for ultrasonic operation at minimum cost. Two such transducers can be driven by one Type 413 generator and several generators can be ganged up to supply any given amount of ultrasonic power.

Cleaning at temperatures from 100–160° F. gives the best results in most cases. The transducers can, however, be operated in liquids at up to 170° F. Work at still higher temperatures can be carried out by confining the hot solution to a separate container and transmitting the ultrasonic energy through an intermediate bath containing cooling coils.

All existing cleaning media can be used in conjunction with ultrasonics. Organic solvents, detergents, alkaline solutions and mild acids can all be employed with these standard transducers. Work in strong acids and corrosive solutions, however, requires the interposition of containers of plastics or glass. When extreme cleanliness is required, the pick-up of dirt from the cleaning solution may be avoided by the use of pre-cleaning and final cleaning chambers.

Processing times cannot in general be quoted, since, for a given component, they depend almost entirely on the economic power input, on the type of dirt involved, and on the degree of cleaning required. As a guide, however, ordinary dirt can be removed in a matter of seconds, while grease rubbed well down between the teeth of a file is cleaned out completely after about 30 seconds' immersion. A pencil mark on the inside surface of a glass sandwich can be removed completely in less than a minute, while a lens would be optically clean in about the same time.



Dawe Type 413 ultrasonic cleaning equipment. Generator left; 1-gal. and ½-gal. tanks on right; separate immersible transducer in front.

Other Applications

Though primarily designed for ultrasonic cleaning, the Type 413 generator can be utilised for experimentation in any field (such as plating, soldering, quenching, pickling, dyeing, emulsifying, metal melting and casting) involving the transmission of ultrasonic energy into a liquid. Experimental results can be applied immediately to full-scale installations by using larger generators and identical transducers in greater numbers.

Metal Finishing Conference

THE Annual Conference of the Institute of Metal Finishing returns this year to Torquay, where the Palace Hotel will be its headquarters from April 15th to 19th. A full and comprehensive programme of technical papers has been planned, and these will be presented for discussion at five sessions on Wednesday, Thursday and Friday. On the Thursday afternoon, a visit to the Corrosion Testing Station at Brixham has been arranged. There will also be ample opportunity for informal social contacts and discussion, in addition to the more formal evening functions, while for the benefit of the ladies attending the Conference a separate programme of entertainment has been arranged.

Change of Address

BRITISH INSULATED CALLENDER'S CONSTRUCTION CO., LTD., announce that the address of their Glasgow Office is now: 110A, Maxwell Avenue, Westerton, Bearsden, Glasgow. The new telephone numbers are: Bearsden 2382/3.

NEWS AND ANNOUNCEMENTS

Institute of Metals Golden Jubilee

THIS year marks the Golden Jubilee of the Institute of Metals, and at the Golden Jubilee Banquet on Thursday, May 1st., The Rt Hon. The Lord Hailsham, P.C., Q.C., Lord President of the Council, accompanied by Lady Hailsham, will be the principal guest, and will propose the toast of the Institute of Metals and the Non-Ferrous Metal Industries.

The Spring Meeting will open on Monday April 28th, when Professor R. S. Hutton, an Original Member and Fellow of the Institute, will deliver the Golden Jubilee Lecture. On Tuesday morning the Annual General Meeting will be held, in the course of which the new President, Marshal of the Royal Air Force, The Rt. Hon. The Lord Tedder, G.C.B., will be inducted, and in the afternoon Professor A. G. Quarrell will deliver the May Lecture on "Fifty Years of Metallurgical Science," which will be followed by a discussion.

Two concurrent sessions will be held on the Wednesday and Thursday mornings and afternoons, and will include a whole-day Symposium on Advances in Inspection Technique as Aids to Production Control in Non-Ferrous Metals Production. Other fields in which papers will be presented for discussion include: age-hardening and related phenomena; the refining of copper and its alloys; the structure and properties of aluminium alloys; chromium; nickel; nuclear fuel elements; and plutonium and techniques for handling it. The meeting will conclude with a series of all-day works visits.

The technical sessions and lectures will be open to non-members as well as to members of the Institute, and tickets of admission will not be required. Attendance at the social functions and visits, however, will be restricted to members of the Institute of Metals and their personal guests and official delegates to the meeting.

Annual Welding Lecture

THE first of a series of Annual Lectures, to be given by eminent scientists and engineers on welding subjects of wide general interest, will be given by Professor Sir Alfred Pugsley, O.B.E., D.Sc., F.R.S., Professor of Civil Engineering at Bristol University, whose subject will be "The Influence of Welding on Structural Design." The lecture will be held in the Conference Hall of the Institute of Welding at 6 p.m., on Wednesday, 12th March. Tea and light refreshments will be available at 5.15 p.m. Tickets will not be required, and a cordial invitation to attend is extended to all who are interested.

The Polarographic Society

A one-day meeting will be held at Ardeer, Ayrshire, on Friday, 26th September, 1958, by kind permission of Imperial Chemical Industries, Ltd., Nobel Division. The provisional programme is as follows:—

The Polarography of Sodium Hydrosulphite, by Professor I. M. Kolthoff.

Polarometric Measurements of Respiration in Biological Systems, by Dr. F. Holton and Dr. R. B. Beechey.

Recent Advances in A.C. Polarography, by Mr. R. Faircloth.

Conductivity Measurement, by Dr. V. S. Griffiths.

Cathode Protection, by Mr. C. L. Wilson.

Some Problems in the Polarographic Analysis of Semiconductors, by Mr. D. R. Curry.

The Use of the Polarograph in Studying Metal Complexes (a) The determination of stability constants, by Dr. F. J. C. Rossotti; (b) The study of some new metal chelating agents, by Dr. T. S. West.

Polarographic Determination of Dibutyl Phthalate in Propellant Compositions Containing Nitroglycerine, by Mr. A. F. Williams.

Arrangements for the meeting, which will include a visit to the Research Laboratory and Works at Ardeer, are in the hands of Mr. A. F. Williams, Research Department, Imperial Chemical Industries, Ltd., Nobel Division, Stevenston, Ayrshire, who will forward details of hotel accommodation, transport arrangements, etc., to those interested. Accommodation will be strictly limited, and anyone desirous of attending should contact Mr. Williams as soon as possible, and in any case not later than 31st May, 1958.

I.B.F. Conference

THE fifty-fifth Annual Conference of the Institute of British Foundrymen is being organised by the East Midlands Branch of the Institute, and will be held at Buxton from May 13th to 16th. The programme opens with a series of works visits in the Derby area on the afternoon of Tuesday, May 13th, and the Conference proper begins on Wednesday morning with the Annual General Meeting and Presentation of Awards, followed by the Presidential Address by Mr. A. E. Peace and the Edward Williams Lecture on "Metallurgy and Aeronautical Progress," by Professor A. J. Murphy. Two simultaneous technical sessions will be held on Wednesday afternoon and Thursday morning and afternoon, whilst Friday will be taken up with a further series of works visits. A special programme has been arranged for the ladies, and in the evenings there will be a number of social functions, culminating in the Dinner-Dance at the Pavilion Gardens on the evening of Friday, May 16th.

Physical Society Exhibition

THE Physical Society's 1958 Exhibition will be held from March 24th to 27th, in the Old and New Halls of the Royal Horticultural Society, Westminster. The size of the Exhibition is expected to be similar to that of last year, and the extra space allowed then for easier access of visitors will be maintained. As is well known, the policy of the Society is to show as many new instruments and techniques as possible, and it is hoped that the Exhibition of 1958 will maintain and exceed its previous high standards. A large number of universities, hospital physics departments and research organizations are again participating, thus assuring a high proportion of purely research exhibits. As is usual, exhibits will show the trend of instrumentation in industry, research and teaching for the next twelve months.

Mechanical Handling Exhibition

THE 1958 Mechanical Handling Exhibition and Materials Handling Convention at Earls Court, London, from May 7th to 17th, is expected to be the largest display of

labour-aiding equipment and methods ever presented to the world. For the first time, the exhibition will be international in character, with over 250 exhibitors displaying time- and cost-saving devices from Great Britain, America, France, Germany, Italy and Scandinavia. At the Materials Handling Convention, running concurrently, leading authorities will present papers dealing with the latest handling methods in particular industries. Further information, details of the many facilities available to visitors, and tickets, can be obtained from the Exhibition Manager, Dorset House, Stamford Street, London, S.E.1.

Personal News

MR. R. OLIVER, who recently resigned his position with P.E.R.A., has taken up an appointment as Chief of Metals Development Department at the Osram Lamp Works, Wembley, and not at the General Electric Co., Ltd., Wembley, as was stated in our December issue.

MR. D. L. CAMPBELL has been appointed Managing Director (Sales) of the Metallurgical Equipment Export Co., Ltd., a consortium of leading companies engaged in the manufacture of steelworks plant.

MR. H. H. BAIRD has resigned his Directorships of Baird and Tatlock (London), Ltd., and Hopkin and Williams, Ltd.

MR. F. J. CLARK has relinquished his appointments with the British Oxygen Group of Companies.

MR. L. H. COOPER, Chairman of The Mond Nickel Co., Ltd., and Vice-President of The International Nickel Co. of Canada, Ltd., has been elected a Director of that Company.

IN order to concentrate under one Director all matters relating to the Steering-Gear Division of George Kent Ltd., MR. L. A. C. BARTLETT, has undertaken the overall supervision of this sector of the Company's activities, and is relinquishing his duties as Commercial Director of the Instrument Division. As a result of this change and to give closer liaison between the Sales and the Research and Development Organizations, MR. R. S. MEDLOCK, has been appointed as Technical and Home Sales Director. He assumes responsiblity for the Contracts Department, Home Sales Representatives and Service and Erection, in addition to his duties as Director in charge of Research and Development. Following a recent survey of the market potential for Kent products in Australia, it has been decided to establish a central organization there under the direction of MR. E. E. COOK, whose position as Home Sales Manager is being taken over by MR. J. THAM. MR. K. SCANES, previously Exhibitions Manager, has taken over from Mr. Tham responsibility for relations with Overseas Subsidiary Companies and MR. W. G. ASKEW, Publicity Manager, has assumed responsibility for the Exhibitions Dept.

MR. J. H. MAYES, General Sales Manager of Northern Aluminium Co., Ltd., has been appointed to the Board of Directors. Mr. Mayes joined the Company in 1934, in the Technical Section of Banbury Works.

MR. W. E. WRIGHT has retired from the Board of Directors of The Pyrene Co., Ltd., and from control of the Metal Finishing Division. MR. H. F. PARSHALL, T.D., who has been on the Board of the Company since 1947, now takes over Board responsiblity for the Metal Finishing Division.

ON appointment as Finance Manager, MR. A. D. SMART has relinquished the post of Secretary, the British Oxygen Co., Ltd. He is succeeded therein by MR. B. R. D. CLARK.

KELVIN and HUGHES (Industrial), Ltd., have appointed MR. V. P. SCHOLES as Area Representative for the Midlands. His address is : "Springcroft," 152, Warren Hill Road, Erdington, Birmingham, 23. (Tel. : Erdington 3856).

MR. L. W. STUBBS, Sales Director, Albright and Wilson (Mfg.), Ltd., MR. A. H. LOVELESS, Technical Director, and MR. A. MC. L. AITKEN, Technical Sales Manager, Kanigen Department, Albright and Wilson (Mfg.), Ltd., recently made a three-week tour of the United States which included visits to Chicago, Los Angeles, Sharon (Penn.), Dunkirk (N.Y.), Buffalo and New York. During the visit, the group had discussions with directors and senior executives of the General American Transportation Corporation, and visited plants operating the Kanigen process throughout America.

MR. E. B. BULL, Works Manager of Wardsend Steel Co., Ltd., has been appointed to the Board of the Company.

MR. G. H. BLAKELEY, Works Manager of Andrews Toledo, Ltd. and Andrews Toledo (Wire Rod), Ltd., has been appointed to the Board of Andrews Toledo (Wire Rod), Ltd. He will continue as Works Manager of Andrews Toledo, Ltd.

MR. J. STRONG, a Director of the British Oxygen Co., Ltd., has relinquished his appointment as Chairman of Quasi-Arc, Ltd., on being appointed Chief Executive Director of British Oxygen Gases, Ltd.

MR. H. HAWORTH has been appointed planning and progress engineer at the Templeborough works of Steel, Peech and Tozer, a branch of The United Steel Cos., Ltd., in succession to the late MR. H. LAWSON.

MR. M. T. CLARK has been appointed Chief Chemist at the Cricklewood Laboratory of British Oxygen Gases, Ltd. He succeeds MR. C. COULSON-SMITH who has retired.

MR. W. T. H. CARTER has been appointed Chief Buyer for the Instrument Division of George Kent, Ltd.

Obituary

We regret to record the death of the following :

MR. W. GEARY, formerly Administrative Works Manager at the Appleby-Frodingham Branch of the United Steel Cos., Ltd., who died in January at the age of 59. Mr. Geary was with Dorman Long and Co., Ltd., before joining Appleby-Frodingham in 1943. Ill-health enforced his retirement in 1955, but he continued with the company as a consultant on management studies. Mr. Geary was a Williams Prize winner of the Iron and Steel Institute, and was Chairman of the Junior Steelmaking Section of the British Iron and Steel Research Association. He was also Secretary of the United Steel Combine's Advisory Committee on Open Hearth Furnaces.

MR. C. FOX PARKER, Sales Manager, of William Mills, Ltd., who died suddenly at his home on Saturday, 18th January, 1958. Mr. Parker joined William Mills as Sales Manager in November, 1955, after some ten years selling for the British Aluminium Co., Ltd.

RECENT DEVELOPMENTS
MATERIALS : PROCESSES : EQUIPMENT

Coke Oven Safety Device

THE W-D gamma interlock is a device which automatically ensures that the charge of a coke oven cannot be pushed out until both doors have been removed and the coke guide to the coke car correctly aligned with the oven. It also incorporates a control, operated from the coke guide or the coke car, whereby the ram-pushing operation can be stopped instantaneously in the event of an emergency.

The device consists essentially of a radioactive isotope mounted on the coke guide in a special holder equipped with a shutter which, when opened, transmits a beam of gamma rays through the space between the top of the charge and the oven roof. The rays act on a geiger-counter mounted on the pusher machine, and this results in the automatic closure of a relay switch which illuminates the green signal light and permits the ram forward travel operation.

The method of operation of the gamma interlock is as follows. The oven doors on both the coke and pusher side are removed in the normal way, and the pusher machine, coke guide and coke car brought into position. When assured of satisfactory arrangements on the coke side, the coke guide operator presses a push button which, by means of an electric thruster actuating through a linkage system, opens the shutter on the transmitter. With the machines correctly aligned and the oven doors removed, the gamma rays pass through the oven to the receiver on the pusher machine, where they are converted into an electrical impulse which closes the relay switch on the ram forward travel circuit, and simultaneously illuminates a green light in the pusher cabin. Only then can the pushing operation commence.

Woodall-Duckham Construction Co., Ltd., Woodall-Duckham House, 63–77, Brompton Road, London, S.W.3.

Multi-Point Control Valves

WHERE a number of main control valves are used in the construction of pneumatically or hydraulically operated machinery, the problem of providing inter-connecting piping for supply and exhaust connections becomes acute, particularly where space is limited, and the pipe size is relatively large. A neat and economical solution can be found by grouping the control valves at a central point, in such a way that these inter-connections are eliminated.

Baldwin Instrument Co., Ltd., have now introduced such a system, which is believed to be unique to the low pressure fluid power field. The valves, which may be fitted with manual, mechanical, electric solenoid or pilot operating mechanisms, are supplied as a multi-point "packaged unit." Any number of valves, up to a maximum of twelve, can be embodied into a single unit. The supply and exhaust ports of all the valves are led into passages running the full length of the assembly, screwed connections being provided in special end pieces, which also provide facilities for mounting the assembly

as a whole. Each end block contains one supply and one exhaust tapping, which are common to all valves. Tappings in each individual valve body provide fluid take-off points for solenoid pilot heads, where these are used. Each main control valve is therefore an entirely independent unit from the functional point of view. This facilitates servicing and also dispenses with separate pilot supply piping. To give an idea of the ultimate economy which can be realised by this system of construction, in the case of a 12 point double solenoid operated hydraulic unit the total number of inter-connecting pipes is reduced from 84 to 2, namely a supply and a drain connection from the pumping set.

At the present time, these "packaged units" are available in the ½-in. bore 4-way pattern only. If any of the points are required for 3-way working only, the unused cylinder connection is plugged. As an extension of this new programme, individual manifold sub-plate mounted valves will be introduced in due course.

Baldwin Instrument Co., Ltd., Brooklands Works, Dartford, Kent.

Smoke Density Control Equipment

PHOTOELECTRONICS (M.O.M.), LTD., are shortly to introduce two new smoke density control units which will have printed circuits throughout. The Type PCSA smoke alarm, suitable for chimneys up to 10 ft. diameter, complies with B.S.2740 : 1956. A light beam projector unit throws a beam of light across the chimney on to a photo-electric cell housed in a receiver unit. When the smoke has reduced the current emitted by the cell to a point set by the operator, a relay closes and the alarm is given.

The Type PCSI smoke density indicator complies with B.S.2788 : 1957, and is suitable for chimneys up to 40 ft. diameter. Operating on the same principle as the smoke alarm, the printed circuit control unit amplifies the varying current emitted by the photocell and this variation is shown on a 10-in. scale meter calibrated in percentage obscuration. Visual and audible alarms are

also incorporated and a variety of recorders can be supplied to give a permanent record of combustion conditions.

Photoelectronics (M.O.M.), Ltd., Fotron Works, 63a, Kingston Road, London, S.W.19.

Temperature Controller

A NEW temperature controller, the Thermal-trol 750, recently introduced by Ether, Ltd., has a wide range of operation, being able to control temperatures between −200° C. and +1,000° C., for electrical, steam, gas- or oil-fired apparatus, with considerable accuracy and speed.

The Type 750 uses as its temperature-sensitive element a platinum wire resistance, or thermo-bulb. The resistance change caused by a change in temperature sets up in an A.C. bridge circuit an unbalanced condition, which is fed into a phase-sensitive amplifier which, in turn, changes the current to the control relay. This system is independent of amplifier gain, and is extremely stable. The control circuit is of a simple design which uses a minimum of components and should ensure trouble-free continuous operation.

The instrument has a 7-in. indicating scale, and is not affected by vibration or ambient temperature: it is suitable for wall or panel mounting.

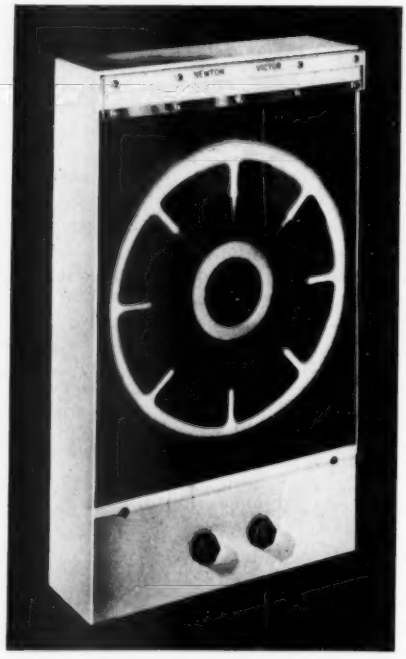
Ether, Ltd., Tyburn Road, Erdington, Birmingham, 24.

Radiograph Illuminator

A FEATURE of the 14 × 17 in. Maxilume illuminators for examining radiographs recently introduced by Newton Victor, Ltd., is the use of 15-watt colour-matching fluorescent lamps giving an evenly diffused, glareless light of true north daylight character, with a complete absence of flicker. These are fitted in an efficient reflector with a special plastics diffuser panel of high light transmission characteristics. The film to be examined is held in position by means of a ball type film holder mounted in a clear Perspex bar, which enables the very top edge of the film to be seen. This feature ensures easy trouble-free insertion, and firm retention of the negative without scratching or marking.

The narrow back to front measurement of 4½ in. is a valuable asset for surface or recessed wall mounting, and the absence of any surround to the viewing panel allows illuminators to be banked side by side with the diffuser panels touching, to form multiple banks of any desired number, with individual control and freedom from dark spaces between viewing panels. If desired, they can also be mounted one above the other. As very little heat is given off by the fluorescent lamps, units may be safely recessed without special ventilation. A removable supporting foot is provided at the back to ensure stability for desk or shelf mounting; keyhole slots in the back allow for wall mounting.

When heavier densities up to 2·5 are to be examined the illuminator can be fitted internally with a high-intensity photoflood lamp so arranged that the fluorescent tubes are switched off when the photoflood lamp is used. A dimming device can also be supplied, in which case an extra control switch is provided on the front panel as shown in the illustrations. The dimming device provides a means of regulating light intensity from full down to 15% of maximum. The rotary control on the front of the illuminator, next to the on/off mains switch, reduces intensity to 75% and 60% in two steps, and then continuously to 15%. The illumination from the viewing panel remains uniform with complete absence of irritating flicker, and " instant starting " is possible at the reduced intensities.



To avoid risk of damaging the wet film by accidental contact with the illuminator, and to prevent water entering the illuminator itself, a one-piece plastic panel can be supplied with the lower end of the panel moulded to form a channel to catch drips from the wet films. The panel is quickly slipped over two chromium plated supports fitted to the upper part of the illuminator. These same supports carry the film hanger, so that the film is held clear of the illuminator panel.

By the use of a special frame mounting, the standard Newton Victor triple roller-blind masking set can be used with the illuminators. By reducing the illuminated area and eliminating the extraneous marginal light, smaller radiographs or particular portions of large radiographs can be viewed and fine detail examined critically without eyestrain.

Newton-Victor, Ltd., 132, Long Acre, London, W.C.2.

Dew-Point Meter

A PRECISION instrument for the measurement of water vapour density and water vapour pressure, proportion of water vapour by volume, and dew-points, is now being manufactured under contract in this country by C. F. Casella and Co. The instrument is suitable for testing in many chemical manufacturing processes, plastics manufacture, air-conditioning and refrigeration installations, and can be employed in the measurement of furnace atmospheres for bright annealing, carburising, carbonitriding and bright-hardening, where the water vapour content of the furnace atmosphere and the atmosphere delivered from the gas generators is of primary importance. In this application, the suitability of an atmosphere is very often assessed in terms of dew-point.

Called the Alnor Dewpoint Meter, this is a portable, self-contained instrument, manually operated and designed for the measurement of the water vapour content of air or any other gas which has a dew-point within the range −80° F. to room temperature. It was developed originally by the Illinois Testing Laboratories, Inc., of Chicago, for the testing of gases that are relatively dry, and therefore within the dew-point range where most other methods of measurement become difficult or impossible.

The apparatus operates on the well-known principle that if a gas is compressed and the pressure released, the consequent expansion results in a cooling effect. If the expansion is sudden, adiabatic conditions are approached, so that the final temperature can be calculated. If the pressure used is such that the gas when expanded cools to a temperature at which the water vapour present begins to separate out as a fog or dew, then this temperature is the dew-point.

By means of a built-in hand pump, a sample of the gas is drawn into the instrument and held in the observation chamber above atmospheric pressure. The temperature of the gas is then read and the pressure quickly released by depressing the operating valve. This also switches the lamp on, illuminating the chamber, and if the gas has cooled to dew-point due to its rapid expansion, a fog will be observed through the window. Even where there are " no dust " conditions, the radium foil incorporated produces ions to act as substitute nuclei around which the droplets can form. The actual dew-point temperature is read from a slide-rule calculator which gives the relationship between initial temperature, pressure ratio and dew-point for the various Q values.

The instrument is claimed to be an improvement on others in this field by eliminating imprecise methods of reading and measurement, by making coolants unnecessary, and by enabling readings to be taken away from the source of the gas. It can be used, within its range, for any gas which is non-corrosive to brass or copper. It is claimed that the instrument has been so designed that it can be operated accurately by semi-skilled personnel.

Electric Resistance Furnace Co., Ltd., Netherby, Queens Road, Weybridge, Surrey.

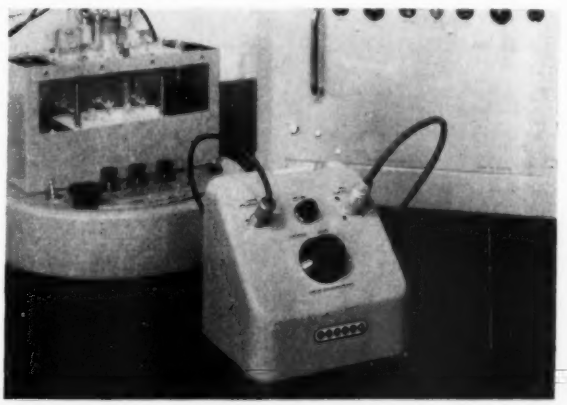
Polarograph Conversion Unit

SOUTHERN INSTRUMENTS Computer Division have recently introduced the K536 anodic conversion unit for use with the K1000 cathode ray polarograph. The latter instrument is suitable for the rapid measurement of polarographic waves due to the reduction of metal ions, or other substances, but waves due to the oxidation of such ions cannot easily be determined. By the use of the K636 unit, measurement of anodic waves is made possible, without affecting the normal presentation of cathodic waves.

The unit comprises a cathodic-anodic switch, a 2V. dry accumulator, together with the necessary connectors to the mercury electrode system and to the cathode-ray polarograph. With the switch in the cathodic position, conditions prevail as in the existing instrument, with the dropping mercury cathode at zero or earth potential. The steadily increasing sweep voltage is applied to the anode in approximately 0·5 V. sections over the range −0·5 V. to +2·5 V. As the cathode is made more negative, any increase in cell current due to the reduction of a particular ion, will be displayed as a peak waveform. The sharp negative pulse arising from the sudden decrease in current when the mercury drop falls off, is used to synchronise the potential sweep to the mercury dropping rate.

If, however, the solution consists of anions undergoing irreversible oxidation, these will be oxidised, under suitable voltage conditions, during the delay period, with the cell current flowing in the opposite direction to the above reduction current. This current will decrease to zero as the cathode potential becomes more negative during the sweep period, and no resulting negative pulse will be provided when the mercury drop falls off, for synchronisation purposes.

In the anodic position, the polarity of the two electrodes is reversed, with the steadily increasing sweep



potential being applied to the dropping mercury cathode. At the same time, the 2 V. accumulator is inserted between the anode and earth, raising the anode potential to +2 V. with respect to earth. Thus the cathode potential may be varied from −2·5 V. to +0·5 V. with respect to the anode, the direction of the voltage change being opposite to that in the cathodic position.

Under these conditions, the anodic waves resulting from the oxidation of the ions, are displayed on the screen of the cathode-ray tube as the characteristic peak waveforms normally presented by the cathodic waves. Synchronisation is achieved as in the previous case, and the potential at which the peak occurs is characteristic of the particular oxidation. The height of the peak is again proportional to concentration.

Only slight modifications are necessary to the K1000 cathode-ray polarograph and the K165 electrode stand for use with the new unit.

Southern Instruments Computer Division, Frimley Road, Camberley, Surrey.

New Uvispek Prism

IMPROVEMENT in the Uvispek spectrophotometer has been effected by the provision of a prism made of synthetic fused silica, which is much more transparent than quartz at wavelengths below 2,500Å. As a result it gives a much better ratio between transmitted and stray light at short wavelengths: it reduces the effect of stray light to negligible proportions at 2,000 Å, and well below. It extends the range of the instrument down to 1,850 Å.

The Uvispek was designed for interchangeable prisms —primarily to gain the advantage of extra dispersion from a glass prism for the visible region—and the new prism can be used with all but the very earliest models of the instrument.

Hilger and Watts, Ltd., 98 St. Pancras Way, Camden Road, London, N.W.1.

Metallic Borides

A COMPREHENSIVE range of metallic borides is now available from Borax Consolidated, Ltd., in two series, one of high chemical purity (not less than 99·8%) and the other comprising technical grades of minimum purity of the order of 99%.

Borides of the more important metals, such as chromium, molybdenum and tungsten, appear in either one or both of these ranges, as do those of some of the less common metals, such as titanium, zirconium, vanadium, niobium and tantalum. The high melting points of metallic borides (up to 3,100° C.) and their hardness make them compounds of major metallurgical interest. Their corrosion resistance, both to noxious gases and to liquid or gaseous metals, has stimulated research into the possibilities of their use in gas turbines and other locations where their inert character is of importance. Many research organisations are investigating fabrication techniques, both from the point of view of pressing by powder metallurgy techniques, and of forming coatings on metals or alloys. The part they play in cermets is well known, and they may also be of use in tools and dies. The eleven compounds in the pure series are dense, hard powders of average particle size of 10–50 microns, and the fourteen in the technical series are finer, averaging 5–10 microns in particle size.

Two technical data sheets—one for each series—have been published by Borax Consolidated, Ltd. These list the chemical analysis, specific gravity, melting point and hardness of each compound. The electrical conductivities, which are of some importance in certain applications, are also included.

Borax Consolidated, Ltd., Borax House, Carlisle Place, London, S.W.1.

Molytone Grease Range

MOLYTONE grease, the combination of bentone grease and molybdenum disulphide introduced some time ago by Rocol, Ltd., has been replaced by a range: Molytone 265, 320 and 380 greases. The original grease was of normal ball and roller bearing consistency—penetration 250–280. It was found to be on the borderline of suitability for application in some grease guns, and too thick for many pressure systems and centralised greasing systems. Also, although bentone greases can be used at temperatures near to the flash point of the oil, continued use at these high temperatures tends to stiffen the grease. Another factor leading to the change was the demand for a Molytone grease suitable for miniature high speed bearings and producing little drag in the very fine clearances of precision machinery.

Molytone 265 grease has a penetration range of 265-280, and is suitable for general grease cups and ball and roller bearings of over 2 in. diameter and of speeds up to 2,000 r.p.m. The next grade, Molytone 320 grease, which is regarded by Rocol as the general purpose grease, has been specially developed for pressure systems, automatic lubricators and ball and roller bearings of under 2 in. diameter and working at speeds of over 2,000 r.p.m. The penetration range is 310–330. The ultra-soft Molytone 380 grease has a penetration range of 370–390, and is intended for speeds of over 10,000 r.p.m., precision work, instruments, servo motors, gyros and all lightly powered instruments. The upper operating temperature for all three greases is 230° C., and the lower limits −20° C., −30° C., and −40° C., respectively.

Rocol, Ltd., Rocol House, Swillington, Nr. Leeds.

Photoelectronic Relay

A NEW light actuated switch produced by Photoelectronics (M.O.M.), Ltd., comprises a relay actuated by a photo-transistor and is suitable for such operations as the counting of articles as they break a light beam; automatic paint spraying; paper breakage alarm; automatic control of conveyors; automatic door opening; loop control; hopper and bunker level control; automatic conveyor jamming detector, and many others. Wherever a light beam can be broken or varied in intensity this unit can be incorporated to perform some automatic control function. The action can be effected when the light beam is broken or when made and time delays can be incorporated if required. Operating from a photo-transistor, no valve amplifier is required, thus making possible the production of a low-cost unit.

Photoelectronics (M.O.M.), Ltd., Fotron Works, 63a, Kingston Road, London, S.W.19.

CURRENT LITERATURE

Book Notice

KEMPE'S ENGINEER'S YEAR-BOOK, 1958

63rd edition. Crown 8vo, 3,000 pages, with numerous illustrations and tables: in two volumes. Morgan Brothers (Publishers) Ltd., 28 Essex Street, Strand, London, W.C.2. 82s. 6d. (plus 2s. 6d. postage).

In its seventy-nine chapters, this 63rd edition of Kempe's Engineer's Year-Book covers practically every branch of engineering, and incorporates a carefully prepared index with over 17,000 references. As in former years, each section and sub-section in the book has received the attention of a specialist in that field, and details of established developments in modern practice are included.

Among this year's additions are the following: "Welding and Cutting" (rewritten to cover argon arc welding, self-adjusting arc welding, controlled arc welding, gas shielded arc cutting, spark machining, oxygen cutting, hardfacing); "Water Engineering" (details of rainfall and catchments, water treatment and plant, reservoirs, dams, water schemes including pumped storage); "Iron and Steel" (revised text on corrosion and protective coatings); "Non-Ferrous Metals and Alloys" (revised with additional text and tables); "Foundry Practice" (revision of diecasting); "Grinding, Abrasives, etc." (details of blasting processes, new abrasive materials); "Machine Tools" (new tables of machine details and text includes cartridge operated hammers and ceramic tool tips); "Wire Ropes" (new text and tables); "Harbours and Docks" (revised text on tides and movement of water, also details of uniform system of buoyage); "Railway Engineering"—permanent way (details of rail fastening devices); "Aerodynamics" and "Aircraft Propulsion" (these two chapters contain revised text dealing with measurement of air speed, wind tunnels, supersonic flight, fuels including high energy fuels, compounded power units, helicopters, parachutes); "Electronic Engineering" (additions to text dealing with oscilloscopes, valves and transformers, including new illustrations); "Atomic Power" (additional text and illustrations); "Heating, Ventilation and Air Conditioning" (revised text and new illustration of charts for pipe sizes, and flow of air in ducts); "Public Health Engineering" (the section dealing with public cleansing has been fully revised); "Concrete and Reinforced Concrete" (revised in accordance with new specifications.

Trade Publications

One of the principal articles in the January issue of *Evershed News*, published by Evershed and Vignoles, Ltd., Chiswick, deals with strain gauges in the aircraft industry, whilst another deals with chromatography recorders. Reference is also made to a new electronic process controller and the latest model of the Evershed polarograph, Mark 19.

A brochure recently issued by Refined Iron Co. (Darwen), Ltd., is devoted to their newly-developed Automat pig iron, which is designed for the production of close-grained castings which are grey in all sections and have good physical properties and machinability. Figures for mechanical properties and tool wear are set out in the brochure, along with the advantages claimed for this material.

Among the large machine tools featured in the September–December, 1957, issue of *The Craven Machine Tool Gazette* are a 27 in. centres roll tenon and wobbler end milling machine, a 15½ in. centres double-ended heavy-type railway axle rough turning lathe, and a 4 ft. railway wheel lathe with special features.

An article describing the automatic ultrasonic inspection equipment developed by Kelvin and Hughes (Industrial), Ltd., and marketed under the name Autosonics, appeared in our May, 1957 issue. The company has now issued a reprint of a paper presented by Mr. H. W. Taylor of the Kelvin and Hughes Research Department at a Convention on Electronics in Automation last June, in which are considered the factors limiting the application of manual inspection under modern high speed production conditions. This consideration is followed by a detailed description of the automatic equipment.

Of particular interest to metallurgical chemists, in the November, 1957 issue of *Hilger Journal* is an article dealing with the fluorite polychromator for determining carbon, phosphorus and sulphur in steel. By this technique the three elements may be determined simply by preparing a sample, pressing a button and waiting a couple of minutes.

In view of the expected increase in demand for direct-arc steel melting furnaces, the English Electric Co., Ltd., has already completed designs for arc furnace transformers as large as 50,000 kVA. and for operation at 66 kV., and the possibility of operating such equipment from the 132 kV. National Grid is being examined. The current (December, 1957) issue of *The English Electric Journal* features a 14-page article on the subject of transformers for this particular application.

In the manufacture of adhesives, careful analytical control ensures a finished product having the correct chemical properties, but the proof of the adhesive is in its performance, and the physical testing of joints made with the adhesive is therefore essential. An account is given in *Aero Research Technical Notes* for January, 1958 (Bulletin No. 181), of the methods and apparatus used by Aero Research, Ltd., in the testing of various types of joint and of structural elements closely representing those found in practice.

The second issue of *Efco-Udylite Review* (February, 1958) contains three articles on technical developments and news and views from the Continent and the Commonwealth. The articles deal with the manufacture of organic brighteners, barrel nickel plating, and the new Efco-Udylite bright nickel processes—the No. 66 family.

Continuing its policy of issuing technical information about the properties, design, finishing and use of diecastings, Fry's Diecastings, Ltd., has recently issued a booklet entitled "Aluminium Alloy Pressure Diecastings. The subject is dealt with comprehensively, consideration being given to diecasting machines, alloys, design features, machining, and finishing processes.

The February, 1958 issue of *Close-Up* features an article on the Roto-Finish process of "precision barrelling,"

equipment for which has been manufactured by Armstrong-Whitworth (Metal Industries), Ltd., since the early 1950s.

THE Alloys Division of Union Carbide, Ltd., has just issued eleven new pages for its loose leaf catalogue of ferro-alloys and electro-metallurgical products. Several of the pages replace sheets in the existing catalogue, but four new products are detailed, namely charge chrome, nitrogen-bearing electrolytic manganese metal, exothermic ferro-manganese, and self-reducing tungsten.

THE November, 1957 issue of *B.T.L. Bulletin*, published by Baird & Tatlock (London), Ltd., contains articles on the Analmatic completely automatic titrator, inorganic analysis with the spectrophotometer, and the I.C.I. sedimentation apparatus for particle size analysis, together with a number of other items of interest to the laboratory worker.

ALTHOUGH essentially batch-type units, bogie hearth furnaces have advantages over the ordinary box-type furnace for certain classes of work. No separate charging machine is required, since the hearth is withdrawn from the furnace for charging and discharging. This type of furnace is thus particularly suitable for handling bulky, unwieldly components which may vary considerably in shape. G.W.B. Furnaces, Ltd., have just issued an illustrated leaflet dealing with electrically heated bogie hearth furnaces.

THE Winter 1957 issue of *Firth Brown News*, the house magazine of Thos. Firth and John Brown, Ltd. and Firth-Derihon Stampings, Ltd., has recently made its appearance. Like its predecessors, it is profusely illustrated —the coloured pictures on the inside covers being particularly attractive—and provides something of interest for the several groups in its varied readership, whether they are engaged in forging boiler drums in Sheffield, or making castings in the Scunthorpe foundry, or handling customers queries in one of the many district offices. Items dealing with the ill-luck which dogged the *Great Eastern*—power production by natural steam in New Zealand, and the Engineering and Marine Exhibition and Farnborough Air Show, are to be found cheek by jowl with the results of the firm's photographic competition and a report of the activities of the works' sports club.

A TECHNICAL DATA sheet giving the physical characteristics, chemical properties and analysis of crystalline boron has just been published by Borax Consolidated, Ltd. The company announced in July this year that they were marketing crystalline elemental boron in the pure state, and the two types dealt with in the technical data sheet show that the purity is as high as 99·15% and 99·6%. The low oxygen and nitrogen content makes the product particularly suitable for incorporation into alloys, and it is expected to be of interest to the electronic and metallurgical industries, and also for certain nuclear applications. Copies of the sheet, No. 5-B, can be obtained from the company at Borax House, Carlisle Place, London, S.W.1.

LEAFLET No. V.51, recently produced by The Incandescent Heat Co., Ltd., deals with the Incandescent-Laclede interchangeable suspended arch, which is intended for use in such high temperature areas as fantail arches and chill arches for open heath furnaces, heating zone arches for reheating furnaces and arches for reverberatory copper furnaces. The new arch permits the use of basic refractories in critical areas and high aluminia tiles in adjacent areas.

ALUMINIUM has given efficient and economical service in heat exchangers for over thirty years, and this application now ranks as a major consumer of aluminium alloy drawn or extruded tube and hollow sections, sheet, strip and plate. A profusely illustrated article discussing the design and manufacture of heat exchangers incorporating aluminium is featured in *The Aluminium Courier* for December last, together with items on the arc-cutting of aluminium, joining aluminium to other metals, aluminium rainwater goods, new welding standards, and applications of aluminium in nuclear engineering.

AN extensive range of industrial and general signs manufactured by a special process is illustrated in a new 40-page booklet produced by Bribond, Ltd. The signs are unaffected by weather or extremes of temperature and will not chip or crack. They are also proof against oil, grease and chemicals, will not corrode, and possess high electrical insulation properties. Since the message is protected under a transparent surface, maintenance is confined to occasional cleaning with a damp cloth.

Books Received

" Complexometric Titrations." By G. Schwarzenbach. Translated and revised in collaboration with the author by H. Irving. 132 pp. inc. index. and 41 diagrams. London and New York, 1957. Methuen & Co., Ltd., and Interscience Publishers, Inc. 21s. net.

" Grain Boundaries in Metals." By D. McLean. (Monographs on the Physics and Chemistry of Materials). 346 pp. inc. author and subject indexes. Oxford, 1957. Clarenden Press : Oxford University Press. 50s. net.

AnalaR Standards for Laboratory Chemicals. 5th Edition. 397 pp. Poole and Chadwell Heath, 1957. The British Drug Houses, Ltd., and Hopkin & Williams, Ltd.

" Aluminium in Building." By E. I. Brimelow. With a foreword by Robert H. Matthew, C.B.E. 378 pp. inc. numerous illustrations, and index. London, 1957. Macdonald and Co. (Publishers), Ltd. 42s. net.

" History of the British Iron and Steel Industry." By H. R. Schubert. Foreword by the President of The Iron and Steel Institute. 445 pp. London, 1957. Routledge and Kegan Paul, Ltd. 60s. net.

" The Structure of Steel." By E. Gregory and E. N. Simons. 176 pp. inc. index, 47 line illustrations and 25 photographs. London, 1957. Odhams Press, Ltd. 18s. net.

" Aluminium von innen Betrachtet." By D. Altenpohl. 200 pp. Dusseldorf, 1957. Aluminium-Verlag G.m.b.H.

" Engineering Properties and Applications of Plastics." By G. F. Kinney. 278 pp. inc. index. New York and London, 1957. John Wiley & Sons, Inc., and Chapman & Hall, Ltd. 54s. net.

Corrosion and Wear Handbook for Water-Cooled Reactors. Edited by D. J. DePaul. 293 pp. inc. index. New York, Toronto, London, 1957. United States Atomic Energy Commission and McGraw-Hill Book Company, Inc. $6.00 or 45s.

Statistics of Non-Ferrous Metals. Production, Consumption, Foreign Trade, Breakdown of Uses—1954–56. 119 pp. Paris, Sept., 1957. O.E.E.C. 12s.

LABORATORY METHODS
MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC
INSTRUMENTS AND MATERIALS
MARCH, 1958. Vol. LVII, No. 341

A New Reagent for the Colorimetric Determination of Aluminium

By H. Green
British Cast Iron Research Association

The disadvantages associated with the use of aluminon for the determination of aluminium are overcome in the procedure suggested here. Aluminon is mixed with ferron in a strongly buffered solution, when a true coloured complex is formed over a wide range of aluminium concentration without any precipitation taking place.

IN the course of developing methods for the analysis of cupola slags at the B.C.I.R.A., it was found that the disadvantages associated with the use of aluminon (ammonium salt of aurinetricarboxylic acid) in the spectrophotometric determination of aluminium, such as the restricted range of aluminium contents for which it is applicable and the need to use a colloidal stabiliser to prevent precipitation of the aluminium lake, could be overcome by using a mixed aluminon-ferron reagent. This reagent gives a strongly coloured complex with aluminium over a wide range of contents without precipitation occurring. The maximum absorption occurs in the region of 520 mμ.

As it is considered that this mixed reagent may find applications in the determination of aluminium in a number of different alloys, general details of the procedure are given in this note.

Aluminium-Ferron Reagent

Determination of aluminium, using a mixed reagent of aluminon and ferron, is carried out on a solution freed from interfering ions such as copper, nickel, iron and titanium. This is achieved by obtaining a solution of the material in perchloric acid and electrolysing with a mercury cathode, when all the heavy metals are deposited. The electrolyte then contains, apart from aluminium, elements such as magnesium, manganese, titanium, calcium and the rare earths. With the exception of titanium, in quantities exceeding that of the aluminium, none of the ions remaining in the solution interfere. Titanium and any remaining traces of iron and copper are removed by extraction with cupferron and chloroform. After destruction of any residue of chloroform or cupferron with nitric and perchloric acid, and dilution, the reagents and buffer solution are added and the colour of the aluminium complex developed by treatment in boiling water for a timed period. After cooling, the extinction is measured on a spectrophotometer, using a blank to set the zero of the apparatus. The heating time is not very critical, and the colour formed is stable for at least twenty-four hours without either precipitation or change in absorption. The ferron solution is quite stable and can be used immediately after preparation. The aluminon solution should be allowed to mature for at least two days before use, and will keep for five or six weeks in a dark bottle away from direct sunlight. Experiments have shown that beryllium is the only element not removed by the procedure outlined which forms a coloured complex with the reagents. The colour formed is such that approximately 4% of beryllium is required to give the equivalent of 1% aluminium.

The calibration graph, which, unlike that for aluminon, is a straight line, is prepared in the usual manner by adding measured volumes of standard aluminium solution, suitable to the range to be covered, to 20 ml. of perchloric acid in a beaker, taking to fumes, diluting to the volume stated and proceeding as detailed below.

Reagents

6% *Cupferron*—6 g. of cupferron stirred for some time with 100 ml. of cold water and filtered just before use.

Ferron Reagent—0·15 g. ferron (7-iodo-8-hydroxyquinoline-5-sulphonic acid) dissolved in water up to 100 ml. in a graduated flask. The solution may be warmed if necessary to effect complete solution.

Aluminon Reagent—1·6 g. aluminon (ammonium aurinetricarboxylic acid) plus 312 g. ammonium acetate and 216 g. ammonium chloride dissolved in water up to 2 litres in a graduated flask.

Buffer Solution—412 g. ammonium acetate and 110 ml. glacial acetic acid dissolved in water up to 1 litre in a graduated flask.

Method

Procedure for 1·5–12·5% Aluminium

A 1 g. sample is dissolved in 20 ml. of s.g. 1·54 perchloric acid (Note 1), and the solution taken to fumes to render any silicon insoluble. The solution is diluted with water and boiled until all the salts are dissolved. (If insoluble material such as graphite or silica is

present, this is removed by filtration). The solution is transferred to a mercury cathode cell and electrolysed for an hour at 3 amp. The mercury is separated from the solution, which is then cooled and made up to 200 ml. in a graduated flask. (Note 2).

A 5 ml. aliquot (≡0·025 g.) is pipetted into a dry 100 ml. separating funnel and 3 ml. of 25% hydrochloric acid and 3 ml. of aqueous 6% cupferron solution are added, each addition being made accurately by pipette or burette. The solution is shaken until any precipitate of titanium and iron cupferrates which may form, has coagulated. 20 ml. of choroform is added and the mixture shaken for a minute or two until there is no colour in the aqueous layer. The chloroform extract is run off and discarded, and the solution shaken twice more with 10 ml. of chloroform, discarding the extracts again. The aqueous portion of the sample is transferred to a dry centrifuge tube and rotated at 2,000 r.p.m. for a minute or two.

A 5 ml. aliquot is transferred from the tube, avoiding any chloroform residue, into a 50 ml. beaker. 2 ml. of perchloric acid, (s.g. 1·54) are added and 2–3 ml. of concentrated nitric acid. The solution is evaporated to fumes of perchloric acid, by which time it should be colourless. If this is not so, a few drops of nitric acid are added and the solution again fumed. The solution is diluted with water while still hot to expel the last trace of nitric acid. 4 ml. of 5% hydrochloric acid are added and the solution warmed if necessary to dissolve any salts. The solution is then cooled and transferred to a 100 ml. graduated flask. 10 ml. ferron reagent, 5 ml. aluminon reagent, and 20 ml. buffer solution are added by pipette and the solution diluted to 100 ml., before being placed in a boiling water bath for a timed period of 5 minutes. Cooling should take place in two stages, the flasks being placed in water at 30°–35° C. for 2–3 minutes, before being placed in cold running water until room temperature is reached, as shown by the liquid level in the flask falling once more to the graduation mark. After cooling, absorption is measured on a spectrophotometer at a wavelength of 520 mμ using a 2 cm. cell and setting on a blank. The blank is prepared by placing 2 ml. of perchloric acid (s.g. 1·54) in a 100 ml. flask, adding 4 ml. of 5% hydrochloric acid, diluting to about 60 ml. and proceeding as in the sample determination.

Procedure for 0·2–1·5% Aluminium

The procedure for quantities of aluminium down to 0·2% is similar to that outlined above. The only difference is that after mercury cathode treatment the solution is made up to 100 ml. in a graduated flask. A 25 ml. aliquot is taken into the separating funnel and 12 ml. each of 25% hydrochloric acid and 6% cupferron solution are added. Three extractions with 20 ml. of chloroform are carried out and, after the aqueous layer has been centrifuged, a 40 ml. aliquot is taken to be fumed down with the perchloric and nitric acids. (Note 3.)

Notes

Note 1.—Samples may be taken into solution directly in perchloric acid, or dissolved initially in nitric or hydrochloric acid and taken to fumes with perchloric acid before electrolysis. Hydrofluoric acid may be used, in conjunction with nitric acid, to obtain solution of siliceous materials, but care must be taken to decompose any fluorides formed, by fuming strongly with the perchloric acid, then diluting with water and taking to fumes again.

Note 2.—With a small sample, such as is taken later in the procedure, if the iron and copper contents are low, as in cupola slags, the mercury cathode electrolysis may be omitted, since the cupferron separation will remove these elements.

Note 3.—Due to a slight variation in acidity when determining the two ranges of aluminium given, it is advisable to calibrate separately.

Acknowledgments

The author wishes to thank the Director and Council of the British Cast Iron Research Association for permission to publish.

Rust-Inhibiting Paint Film

Dramatic shots of the damage caused by rusting open a 22-minute new colour film, featuring Rustodian Rust-Inhibiting Calcium Plumbate Paint, a product of Associated Lead Manufacturers, Ltd., from whom copies of the film may be borrowed. A flashback to 1941 shows a reconstruction of the experiments leading to the discovery of the rust inhibiting properties of calcium plumbate, and the subsequent work involved in developing the pigment and the paint.

Like red lead, calcium plumbate provides protection by a combination of basic and oxidising characteristics, as well as film-forming properties by interaction with linseed oil. It is believed to passify both cathodic and anodic areas, on iron and steel, and in this respect it differs from other rust-inhibitive pigments. It is also believed to be unique among primers in that it can be applied to newly galvanised surfaces without preliminary etching. Animated diagrammatic sequences are used to explain the mechanism of corrosion protection, and a series of shots illustrates the use of Rustodian as a primer with almost universal application.

Other sections of the film show the manufacture of Rustodian, using Caldiox pigment, and the tests to which it was subjected in the Shetlands, where the salt-laden atmosphere is amongst the most corrosive in the British Isles. Finally, the film shows the use of Rustodian in various fields, including building and marine engineering.

Yorkshire Imperial Metals, Ltd.

Yorkshire Imperial Metals, Ltd., the new company which represents the fusion of the copper and alloy tube, fittings and plate activities of the Yorkshire Copper Works, Ltd., and of Imperial Chemical Industries, Ltd., disposes of assets worth about £18 million, including the former I.C.I. plants at Kirkby (Liverpool), Smethwick (Staffs.), Landore (Swansea), and Dundee, and the former Yorkshire Copper plants at Leeds, Barrhead (Glasgow), and Castleford (Yorks.). The registered office of Yorkshire Imperial Metals, Ltd., is at Haigh Park Road, Leeds.

The Board of Directors has been constituted as follows: from I.C.I.—Dr. J. Taylor (chairman), Mr. P. T. Menzies, Dr. M. Cook, Mr. M. J. S. Clapham, Mr. St. John Elstub, Mr. H. Royle, and Mr. W. N. Ismay; from Yorkshire Copper—Mr. G. P. Norton (deputy chairman), Mr. H. F. Sherbourne (managing), Mr. W. R. D. Macdonald, Mr. C. G. Robinson, Mr. D. Fraser, Mr. J. Christie, and Mr. C. Breckon. The company's secretary is Mr. P. D. Peel Yates.

The Metallographic Application of X-ray Scanning Microanalysis

By D. A. Melford, M.A., Ph.D.* and P. Duncumb, M.A., Ph.D.†

A scanning version has been developed of the Castaing technique for determining the composition of a minute area on a metal surface by irradiating it with a fine focus electron beam and analysing the resultant X-ray emission. The authors demonstrate here the close correlation between results obtained with the new instrument and conventional optical micrographs.

Introduction

MANY metallurgical difficulties arise as a result of segregation, particularly on the micro-scale. In certain cases where actual precipitation has occurred, elegant extraction replica techniques are now available to the metallographer which help him to isolate and identify the precipitate particles. Solution segregation, on the other hand, is still a somewhat less tractable phenomenon.

The technique of irradiating a metal surface with a fine focus electron beam and analysing the resultant X-ray emission, originated by Castaing,[1] is being currently developed at several centres. By this means it is possible to make, within certain limits, a qualitative and quantitative analysis of a volume at the surface of the metal approximately hemispherical in shape and about 1 micron in diameter. This comparatively high resolution makes possible the detection of highly localised fluctuations in segregation. The existence of such regions, frequently suggested in the past, has hitherto been impossible to demonstrate satisfactorily in practice, owing to the low resolution of the available sampling and analysis techniques.

The full exploitation of this development as a metallographic tool depends not only on the facility with which these regions of high positive (or negative) segregation can be located in the specimen, but also on the ease with which the results can be correlated with optical micrographs. It is felt that the apparatus described below has several advantages in this respect.

Description of Apparatus

The scanning technique[2-4] has been developed by one of the authors (P.D.) in collaboration with Dr. V. E. Cosslett of the Cavendish Laboratory, Cambridge. An area approximately $\frac{1}{2}$ mm. square on the surface of the specimen is scanned by an electron beam 1 micron in diameter, and the X-ray emission is collected by a counter. The output from the counter modulates the brightness of a beam scanning an oscilloscope screen in synchronism with the electron beam scanning the specimen. By means of a crystal spectrometer it is possible to select one particular X-ray line and collect in the counter the emission at this wavelength only. An image is thus formed in which the brightness at any point is proportional to the amount of the emitting element present in the corresponding point in the surface of the specimen. Scanning can then be stopped and the probe positioned (by reference to the displayed image) on any interesting feature. Quantitative analysis is then carried out by recording the intensity of the emission on a pen recorder, and comparing with the intensity of the emission from a specimen of the pure element when irradiated at the same beam current. Alternatively, average analyses can be obtained over any desired region by appropriately limited scanning.

Under the present operating conditions, electron diffusion in the specimen limits the resolution to about 1 micron, so that the maximum useful magnification of the X-ray image on the oscilloscope screen is approximately × 1,000. There are also facilities, however, for collecting the high energy scattered electrons by means of a scintillation counter and again displaying an image on the oscilloscope screen. In this respect the instrument functions as a scanning reflection electron microscope, and it is thus possible to examine the surface topography at considerably higher resolution.

With slight modification, the instrument would be suitable for the analysis of all elements heavier than chlorine ($Z = 17$) and possibly silicon ($Z = 14$). Major changes in the recording system would be needed to extend the range to carbon.

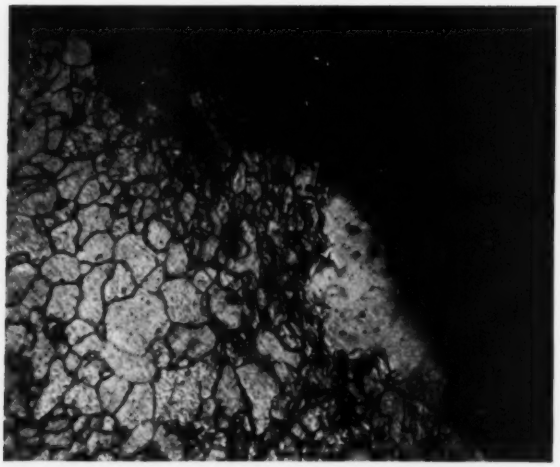
Experimental Results

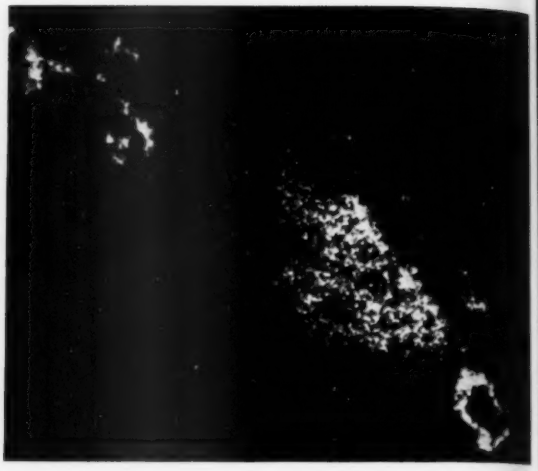
The potentialities of this technique are well illustrated by the accompanying photographs, taken during an investigation of the hot-shortness exhibited by the surface of a mild steel specimen. The generally accepted view of this phenomenon is that a build-up in the concentration of elements such as nickel, copper and tin occurs at the base of the scale when steel containing these elements is heated in an oxidizing atmosphere, due to the selective removal of iron in the oxidation process. This can lead, particularly if tin is present, to the formation of material which is molten at normal hot working temperatures. The segregation of tin is thus of particular interest in this case, and a comparatively trivial modification of the present apparatus will enable this to be determined in a manner similar to that described below for nickel and copper. The percentage chemical composition of this steel was given as :—

C	Si	Mn	S	P	Ni	Cu	Sn
0·10	0·07	0·51	0·049	0·018	0·14	0·20	0·059

Fig. 1 is a conventional optical micrograph (× 200) showing two small surface cracks and a large " inclusion " —metallic in appearance. This is compared with Figs. 2, 3 and 4, taken at the same magnification in the X-ray scanning microanalyser with Ni Kα, Cu Kα, and Fe Kα radiations respectively selected. The segregation of nickel, and to a lesser extent copper, in the neighbourhood of each crack and in the large inclusion are clearly demonstrated by Figs. 2 and 3, while Fig. 4 exhibits the

* Tube Investments Research Laboratories, Cambridge.
† Cavendish Laboratory Cambridge.





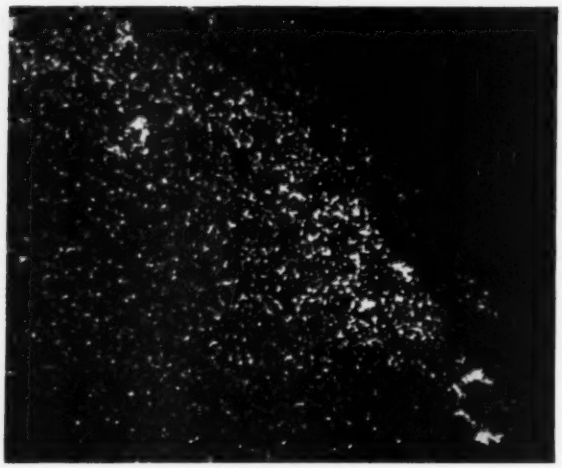


Fig. 1—Optical micrograph ×200
Fig. 2—Ni Kα radiation. ×200
Fig. 3—Cu Kα radiation. ×200
Fig. 4—Fe Kα radiation. ×200

relative depletion of iron in areas enriched in the other elements.

Fig. 5 is again a conventional optical micrograph showing the smaller of the surface cracks in Fig. 1 enlarged to × 600. Figs. 6, 7 and 8 are the corresponding X-ray micrographs taken at the same magnification by selecting Ni Kα, Cu Kα, and Fe Kα radiations as before. The segregation of nickel and copper is again well demonstrated in the region surrounding the crack.

Average quantitative measurements made by scanning an area located wholly within the large " inclusion " led to values of 6% and 2½% for the nickel and copper contents, respectively. Two particularly bright spots are visible in the inclusion area on closer inspection of Fig. 3, one near the surface and one a small distance below it. The probe was centred in the latter region, and the results obtained showed that the nickel and copper contents had here risen to 22% and 7%, respectively. Movement of the probe a distance of 2–3μ in any direction from this position, however, led to a fall in the recorded concentrations to something approaching the average figures previously obtained. Somewhat greater segregation was observed in parts of the wall of the smaller crack. Analysis of one of the brightest regions of Figs. 6 and 7 led to values of 33% Ni and 21% Cu.

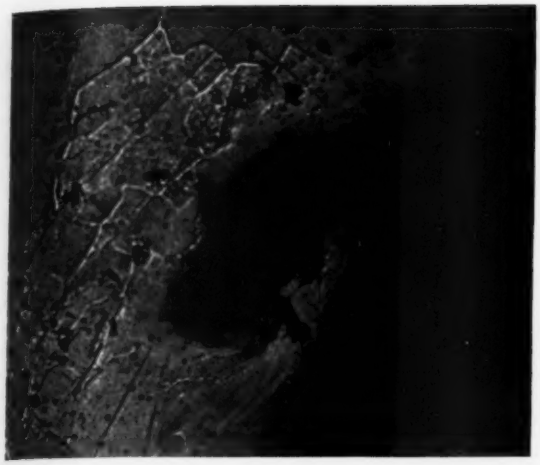
For comparison with these results, a small area well below the surface was scanned, and the nickel and copper contents in the bulk were both found to be below the sensitivity limit at 0·4%. This limit can be reduced by lengthening the recording time and increasing the beam intensity; in practice a figure of 0·02% has been obtained.

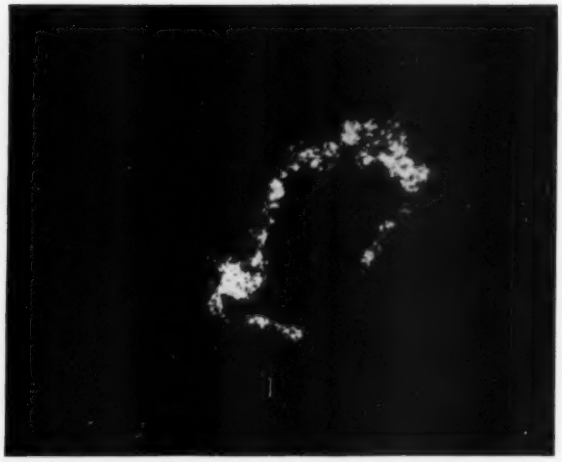
The quantitative results may thus be summarised as follows :—

	Ni%	Cu%
Bulk chemical analysis	0·14	0·20
Microanalysis of steel below surface ..	<0·4	<0·4
Microanalysis of large inclusion .. .. (average by scanning)	6	2·5
Microanalysis of brightest spot in large inclusion (2-3μ in diam.)	22	7
Microanalysis of brightest spot in crack wall	33	21

The error in these estimates is of the order of ±5% on the highest values and less on the lower. This can be reduced still further by applying certain corrections.

It is probable that no other technique at present





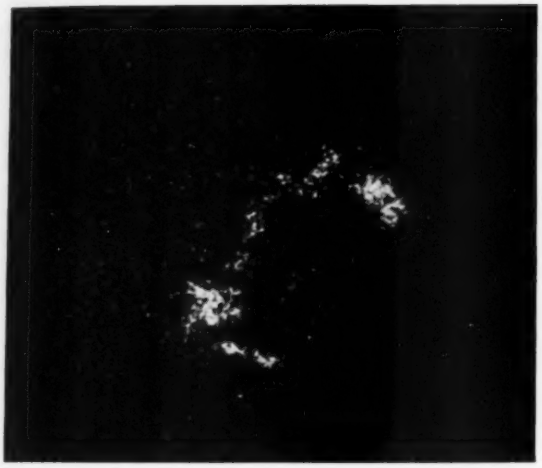


Fig. 5—Optical micrograph. ×600
Fig. 7—Cu Kα radiation. ×600

Fig. 6—Ni Kα radiation. ×600
Fig. 8—Fe Kα radiation. ×600

developed is capable of demonstrating and determining such highly localised peak fluctuations in solute concentrations with equal facility, and it is felt that the instrument will be of considerable service to the metallographer in this respect.

Future Development

A new instrument designed specifically for metallographic work is now under construction at the Tube Investment Research Laboratories. Facilities are being provided for optical observation of the specimen inside the instrument in order to assist location of the field of interest. The specimen is then positioned under the electron beam in such a way that this field is automatically re-located and the reflection electron image at the same or greater magnification is examined before proceeding to the X-ray image. A multi-position specimen stage will enable several pure metal standards to be moved in succession under the electron beam, and thus permit rapid calibration for the quantitative measurements.

Acknowledgments

The work has been made possible by D.S.I.R. awards to one of us (P.D.), and our thanks are also due to the Chairman of Tube Investments, Ltd., for permission to publish this paper.

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Dow International Communications

A NEW communication channel linking The Dow Chemical Company's overseas sales offices and subsidiaries in Europe with the Company's headquarters and plant locations in the United States was opened on February 10th. Replacing the previously used teletypewriter system, a new radio circuit between New York and Zürich, Switzerland, has been leased by the company from R.C.A. Communications, Inc., and Radio-Suisse. This marks the beginning of a planned, fully integrated and privately operated network connecting Dow, Dow Chemical International Limited and Dow Chemical Inter-American Limited throughout the world. The two latter organizations are export subsidiaries of Dow.

Some Observations on the Measurement of Spectral Line Intensities by Microphotometry of the Photographic Plate

By D. R. Curry, B.Sc., A.R.I.C.

*Bragg Laboratory, Naval Ordnance Inspection Department, Sheffield**

IN the course of a project concerned with the application of direct reading to a large grating spectrograph, preliminary work using a photographic plate involved comparative intensity measurements. Time did not permit an exhaustive study of such procedures, but the following examination of microphotometry may be of general interest.

The accuracy of intensity measurements by microphotometry of the photographic plate has been the subject of many papers.[1-4] The work to be described forms a comparative link between some of the instruments previously studied, and also notes a property of the characteristic curve when determined by normal procedures.

Consideration of the procedure for determination of the "log. ratio" of a line pair shows that it may be broken down into stages, as follows:

(*a*) Evaluation of the characteristic curve for the emulsion under the conditions of measurement.

(*b*) Measurement of the densities of the two lines.

(*c*) Conversion of the densities (*b*) to log. RI values from the characteristic curve (*a*).

(*d*) Obtaining the difference between the two (*c*) readings to give the log. ratio.

Experimental

All spectrograms studied were taken with a Jarrell Ash 3·4 m. spectrograph.[5] This Wadsworth mounting of a grating gives a stigmatic image of high quality, with a maximum length of line (uniform illumination) of 20 mm., permitting the use of a rotating step sector for plate calibration purposes. The sector had eight steps, each approximately 2mm. deep with ratios log. $(I_n / I_{n+1}) = 0{\cdot}2$. Two microphotometers were used: the Zeiss, described by Stasheim,[1] and the Hilger, which was used by Shirley.[4] In the present work, the rotating step sector was used for calibration of the emulsion, although it has been stated that this is not the most reliable procedure.[6]

The first examination was to determine the relative accuracy of the two microphotometers for measuring the photographic density. In each case five sets of fifteen lines were selected at random in different regions of a plate (throughout this work Kodak B10 emulsion has been used). Each set of lines was read three times, the only movements between readings being those of the microphotometer plate carriage to traverse the line across the slit and move the plate to different spectrograms. The triplicate readings obtained were then analysed to give the standard deviation of the density measurement in terms of density units. In the case of the Zeiss, the 100 cm. density scale was read direct, giving a value for σ of 0·0015. With the Hilger instrument, the 50 cm. linear scale was read and converted to density in the normal manner, to give a value $\sigma = 0{\cdot}0031$. In both cases, the slit used was equivalent to the initial spectrographic slit at the magnification of the microphotometer. It is considered that the improvement in performance between the two instruments is partly due to the increased scale length and the heavier and more stable construction of the Zeiss plate carriage. The remainder of the work described was carried out using the Zeiss instrument.

Five further sets of fifteen lines with densities ranging from 0·05 to 0·7 were selected, and their densities read at the same time as the characteristic curve for the emulsion was determined by reading the steps of a step sector spectrogram in the same spectral region. These readings were also triplicated, but on different occasions. In each case the characteristic curve was re-evaluated to compensate for imperfect alignment of spectrum lines with the microphotometer slit when the plate is initially placed on the carriage. The characteristic curves were drawn on 30 in. square millimetre paper, and the density readings obtained converted to log. RI units. By the same analysis procedure as before, a standard deviation was evaluated for this stage at $\sigma = 0{\cdot}0029$ log. RI units.

It was then possible to study the accuracy of determination of a "log. ratio" from the above figures. Pairs of lines were selected and the differences in their log. RI values (i.e. the log. ratio) compared on the three occasions of measurement. This process showed a σ of 0·0030 log. RI units. Consideration of this value suggests a certain compensation of the graphical errors arising in determining log. RI when a log. ratio is found. The range of log. ratio values was between 0 and 0·8.

Discussion

Comparing this work with that of previous authors[3,4] the values obtained sustain that of Shirley[4] on the Hilger instrument, although he ascribes the difference between the reading error and total error of a log. ratio to small scale plate variability. In comparison with Sawyer and Vincent's results,[3] however, some disagreement is still apparent, but their procedure in determination of reading error may be questioned. In reading a 50 cm. linear scale, deflections were only estimated to the nearest 0·05 to give a σ of 0·027 cm. In the present work, the 100 cm. density scale was read to 0·0002 density units whenever possible, giving a standard deviation of 0·0015. If similar approximations had been made in reading, the final value for the reading error would compare favourably with that obtained by Sawyer. Whilst in practical use there is no need to read to the precision attempted here, it is felt that accuracy in the overall spectrographic determination, as opposed to reproducibility, justifies closer reading of a 50 cm. scale than half a millimetre.

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Reliability of the Characteristic Curve Evaluation

It has long been known that when comparing readings of lines of different spectral sharpness it is vital to specify the microphotometer slit width, when using the normal technique of reading a peak density value as the line is slowly traversed across the slit. As a preliminary to the direct reading project, where the total intensity of a line is measured by photomultiplier, some observations were made to compare total density with peak density measurements. A number of spectral lines of varying sharpness in the same region of a step sector spectrogram were selected. Using a narrow microphotometer slit width and the linear scale, each of these lines on each step was traversed and readings taken at 0·01 mm. intervals. The results were plotted on semi-log paper to give a density distribution curve and the areas contained determined with a planimeter. The areas were thus a function of " photographic total density " and could be compared with the peak densities read from the curves. Characteristic curves for the emulsion were then drawn for peak density/log. RI and total density/log. RI for each line. Somewhat contrary to expectation, it was found that the characteristic curves obtained by the peak density method were superimposable, although obtained from lines of widely differing spectral quality. The " total density " characteristic curves, however, varied in slope and curvature with the nature of the spectral line.

The result, which fully justifies the present method of measurement, is presumably explained by the fact that any particular step, using the total density method, involves a range of densities which will in themselves be dependent on the characteristic curve. Thus any direct total density/log. RI relationship is complicated, if not masked completely.

Acknowledgments

The author wishes to thank Mr. K. Oldfield for experimental assistance and Mr. J. H. Oldfield for encouragement in this work. This paper is published by permission of the Admiralty.

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Laboratory Arc Furnace

Vacuum or Inert Atmosphere Melting

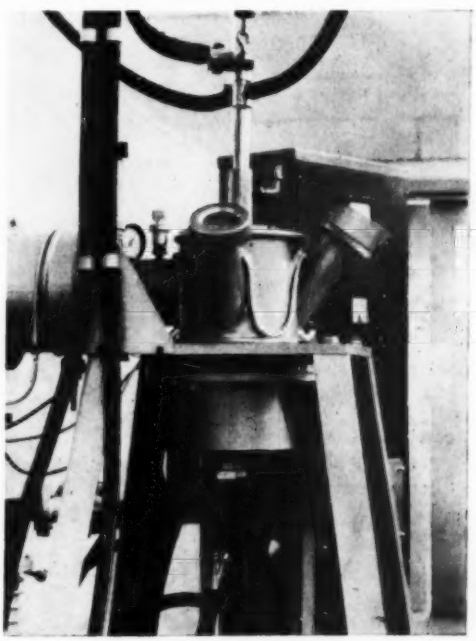
THERE is great interest in the metallurgical industry at the moment in all aspects of vacuum melting, and the use of vacuum-arc and argon-arc furnaces is of considerable importance. Refractory metals such as molybdenum and tungsten require the attainment of high temperatures for melting, and this can be achieved by using a direct arc as the heat source and a water-cooled copper hearth as the crucible material.

Laboratory scale arc furnaces designed for this class of work are now being produced by Edwards High Vacuum, Ltd., of Crawley. The first model was delivered to the Technical University of Trondheim, Norway, last year, and since then several other production models have been delivered to a number of centres in this country. The Edwards furnace is capable of being used for both consumable and non-consumable electrode work, according to requirements.

Two Modes of Operation

In non-consumable electrode operation, a non-consumable electrode of tungsten is held in a water-cooled copper stem, which is externally connected to a D.C. generator. The positive pole of the generator is connected to a flat-bottomed water-cooled copper hearth. Using this arrangement, specimen buttons of high purity may be prepared in a low pressure argon atmosphere, temperatures in the region of 4,000° C. being attainable: e.g. tungsten and molybdenum buttons may be readily prepared from sintered pieces of the metal. This technique can also be adapted for making finger type ingots, by replacing the usual flat-bottomed hearth by one shaped to permit casting of this type of ingot in a horizontal plane.

In the other technique, a consumable electrode of the metal or alloy to be melted is held in a water-cooled feeding stem, which is externally connected to the negative pole of a D.C. generator. A hearth is formed by a water-cooled copper tube connected to the positive pole of the generator. This arrangement is suitable for producing ingots up to 5 lb. steel capacity, and with this method melting can be carried out in a continuously



Close-up of the furnace body, showing the arrangement for non-consumable electrode operation.

evacuated system. In general, the use of this system extends up to temperatures in the region of 2,600° C., and it is expected that considerable work will be carried out, not only on reactive metals such as titanium and zirconium, but also on conventional metals and alloys, as these too may be cast in the arrangement provided. Working temperatures in excess of 2,600° C. may be attained by this technique, but a change in generator characteristics and water-cooled devices is necessary.

Construction

Copper plated mild steel is used for the construction of the furnace body, which has an internal diameter of $7\frac{1}{4}$ in. and a height of $6\frac{1}{4}$ in., and is water-cooled by means of copper pipe soft soldered to the outside. Projecting from the body are two sighting ports, a rough pumping port, and a high vacuum pumping port. The body is flanged top and bottom for the attachment of the alternative electrode and hearth assemblies.

In the non-consumable electrode system, the hard chrome plated copper tube carrying the electrode is fitted via a universal vacuum seal to a water-cooled flange dimensioned to mate with the body flange. This arrangement enables the electrode to be moved in any direction and made to touch any part of the hearth.

The consumable electrode feed mechanism consists of a water-cooled copper rod which enters the main vessel through a double "O" ring seal and is raised and lowered by means of twin rotating shaft movements attached to its upper end, which is external to the vacuum system. The rotating shafts are hand operated by means of a chain drive and a handwheel conveniently situated for ease of operation whilst viewing the furnace interior. The electrode itself is attached to the lower end of the copper rod by means of a copper collar and grub screws, whilst the power connection is made direct to the copper rod with flexible cables. The complete assembly is fitted on to a water-cooled flange dimensioned to mate with the furnace body flange.

The mould for the consumable electrode set-up consists of a $\frac{1}{8}$-in. thick copper cylinder, $1\frac{3}{4}$ in. internal diameter $\times$ $10\frac{1}{2}$ in. long, closed at the bottom and secured at the top to a special water container, which is itself secured to the base of the furnace body. A rapid turbulant water stream surrounds the mould and ensures effective cooling. A coil wound round the outside of the water jacket to cover the length of the mould is available for the production of a magnetic field to secure mixing of the molten metal and stabilisation of the arc.

The vacuum seal on the mould is so designed that should the seal break down through excessive heating, air and not water enters the furnace, thus greatly minimising the risk of a hydrogen explosion resulting from the dissociation of the water.

Pumping System

The dual purpose version of the furnace is pumped by a 9B3 booster vapour pump backed by a 1SC450 rotary pump displacing 15 cu. ft./min. These vapour booster pumps have been specially designed for metallurgical processes where large-scale degassing occurs during heating and melting. A special feature of the 9B3 is its ability to work against backing pressures as high as 3 mm. Hg, thus enabling it to be used in the pumping cycle at a point where the volumetric efficiency of the rotary pump is still high. The unbaffled speed of this pump is 900 litres/sec. at working pressures as low as 0·004 mm. Hg, and it has a maximum gas throughput of 18,000 litre microns/sec.

Where the furnace is required solely for non-consumable electrode operation, the high speed booster pumping system is not necessary, as the furnace will operate in a low pressure argon atmosphere. To maintain purity in melting, however, a small pumping system is provided consisting of a model 203 oil diffusion pump capable of attaining an ultimate vacuum of 5×10^{-6} mm. Hg, and having an unbaffled speed of 80 litres/sec. This is backed by a Speedivac 1SC150 rotary pump displacing 5 cu. ft./min. Vacuum measurement in the backing line is provided by a B2 Pirani type vacuum gauge, which can also be used as a leak detection unit using the hydrogen/Pirani method.

The furnace is normally intended to be operated from a 600 amp. dual continuous control arc welding generator, although this may vary according to the nature of the work for which the furnace is to be used. The plant is completely self-contained, and occupies a floor space of 4 ft. $\times$ 4 ft. with a height of 9 ft. Vacuum gauges and other electrical instruments are built into a separate pedestal cabinet.

Data Handling System for Capenhurst

IMMEDIATE and accurate display of operating information is essential to the efficient control of every chemical process. To improve this particular aspect of the plant for gaseous diffusion of uranium hexafluoride at Capenhurst, the United Kingdom Atomic Energy Authority has awarded to Sunvic Controls, Ltd., an A.E.I. company, a contract for a data handling system which, as envisaged, will be the largest in Europe, both as regards value and number of points scanned.

There will be eight units each capable of scanning 260 points measuring process gas, gland and motor bearing temperatures, as well as cooling water temperatures, motor currents and voltage. Each unit will monitor signals from a group of four "cells." In a central control room will be equipment for printing, on demand, a tabulated log showing the value at all measuring points on any one of the 32 cells, the cell identification and the time of scanning. Any point exceeding pre-determined limits will be shown on the log sheet in red. Fitted to each of the eight logger units will be an alarm printer to record the time, identification and value when any point goes "off-normal" or returns to normal; once again "off-normal" values will be shown in red, and there will also be a code to show whether a point is high, low or defective.

Additionally, two mobile print-out units will be supplied. These will enable a complete log to be obtained at any time, for any cell, without interrupting normal scanning of the other three cells in the group.

Changes of Address

NEW METALS & CHEMICALS, LTD. and Fleischmann (London), Ltd., announce their removal to larger premises at: Chancery House, Chancery Lane, London, W.C.2. Their telephone numbers have been changed to HOLborn 7415 (8 lines) whilst their teleprinter and telegraphic address remain as before.

ON and after March 1st, 1958, the address of the Midlands Office of Wild-Barfield Electric Furnaces, Ltd., will be: 71 Broad Street, Birmingham, 15. The telephone number will be Midland 7232.